

PARTICLE SIZE ENLARGEMENT

C.E. CAPES

*National Research Council of Canada,
Ottawa, Canada*



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HANDBOOK OF POWDER TECHNOLOGY

Edited by **J.C. WILLIAMS** and **T. ALLEN**

*School of Powder Technology, University of Bradford,
Bradford, West Yorkshire, England*

Vol. 1 Particle Size Enlargement (C.E. Capes)

Vol. 2 Fundamentals of Gas-Particle Flow (G. Rudinger)

Vol. 3 Solid-Gas Separation (L. Svarovsky)

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School of Powder Technology, University of Bradford, Bradford, West Yorkshire, England

The Handbook will present, in convenient form, existing knowledge in all specialized areas of Powder Technology.

Information that can be used for the design of industrial processes involving the production, handling and processing of particulate materials so far does not exist in a form in which it is readily accessible to design engineers. Scientists responsible for characterizing particulate materials, specifying the requirements of industrial processes, operating plants, or setting up quality-control tests all have similar problems in their fact-finding missions through the scattered and scanty literature. The aim of this handbook is to remedy this deficiency by providing a series of thematic volumes on various aspects of powder technology. Each volume is written as a monograph and can be used independently of other volumes.

Emphasis will be placed on setting out the basic concepts of the subject and discussing their applications to the design, selection and operation of equipment of an industrial scale. To ensure timely publication, each volume will be published as soon as the material has been delivered by the authors.

Titles published or in production:

Vol. 1: Particle Size Enlargement (C.E. Capes)

Vol. 2: Fundamentals of Gas-Particle Flow (G. Rudinger)

Vol. 3: Solid-Gas Separation (L. Svarovsky)

Forthcoming titles:

Particle Size Measurement (T. Allen)

Measurement of Surface and Pore Size of Solid Particles (T. Allen)

Filtration (A. Rushton)

Design of Solid Handling Plant (J.C. Williams)

Pneumatic Conveying of Solid Particles (J.S. Mason et al.)

Mixing and Segregation of Solid Particles (J.C. Williams and N. Harnby)

Mechanical Conveying of Solid Particles (H. Colijn)

Powder Coating (J.F. Hughes and S. Singh)

Dust Explosions (P. Field)

Fineparticle morphology (B.H. Kaye)

EDITORS' PREFACE

This monograph on size enlargement of particles is the first of a series which will together form a Handbook of Powder Technology, primarily intended for engineers and scientists working in industry.

The scope of the Handbook can be defined as being concerned with that part of chemical engineering which deals with processes involving the handling and treatment of material in solid particulate form.

Such a Handbook requires little justification. The characterisation and behaviour of particulate systems are largely neglected in the education of engineers and scientists. Courses in chemical engineering, in particular, are almost entirely concerned with fluid systems, yet the graduate engineer or scientist will frequently find that industry presents many problems in the behaviour of particulate material for which his or her previous education has given a quite inadequate preparation. The aim of the handbook is to remedy this deficiency by providing monographs on various aspects of powder technology. It is also hoped that by providing suitable texts the teaching of powder technology in universities and polytechnics will be encouraged.

Each monograph will be written by an expert in the particular aspect of the subject covered and will be published as soon as it is prepared. This avoids the problems associated with large multi-author books; the information will be available in a more convenient form and the up-dating of various parts of the Handbook will be facilitated.

Comments and suggestions for improving the Handbook, as well as proposals for further titles, would be welcomed by the Editors.

University of Bradford
March, 1980

J.C. Williams
T. Allen

AUTHOR'S PREFACE

The methods used to create larger entities from fine particles so that the bulk properties of particulates can be improved is the subject of this book. These so-called "size enlargement" methods evidently concern a broad spectrum of technical disciplines and industries ranging from the relatively small scale requirements of pharmaceutical manufacturers through the tonnage requirements of the fertilizer and minerals processing industries.

A primary objective in preparing this book was to present a generalized account of the many size enlargement techniques scattered throughout these diverse industries, with emphasis on similarities and unifying characteristics whenever possible. A related objective was to allow the reader to understand the underlying principles so that successful techniques from other industries can be adapted to the application of his particular concern.

This book is part of a series forming a Handbook of Powder Technology. By definition, a handbook should be concise. To this end, information is presented in tables, diagrams and figures whenever possible. Descriptive text is kept to the minimum felt to be necessary in understanding the subject with emphasis on the equipment used and its operation.

In organizing this treatment, it was decided to devote one chapter to each of the principal methods used to bring particles together into agglomerates, viz.:

1. Agitation methods — tumbling agglomeration
2. Agitation methods — mixer agglomeration
3. Pressure methods
4. Thermal methods
5. Spray and dispersion methods
6. Agglomeration from liquids

These six topics, together with introductory material (Chapter 1) and considerations on agglomerate strength (Chapter 2) comprise the eight chapters of the book. Readers looking for information on a specific size enlargement technique can refer directly to the appropriate chapter. If the problem is to find a suitable technique for a new application, however, a preliminary selection procedure is outlined in Chapter 1.

In preparing a concise account, it has often been necessary to delete theoretical background and other materials that would be included in a larger book. By way of compensation, a quite comprehensive list of references has

been included with each chapter for readers wishing to obtain a greater depth of knowledge.

At the time of writing, conversion from the Imperial (F.P.S) to metric system of units, especially in North America, is far from complete. Thus Imperial units have generally been used in the text with metric equivalents provided in brackets. In tables and figures, the system of units given in the original source (usually Imperial) has been retained.

The author gratefully acknowledges the many individuals on the scientific, technical, secretarial and librarial staff of the National Research Council of Canada who assisted in and supported the preparation of this book.

The cooperation of those in industry and elsewhere who provided information for this handbook and/or allowed copyright material to be reproduced is also acknowledged. References to these sources are given in each chapter.

Ottawa, Canada
January, 1980

C. Edward Capes

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Chapter 1

INTRODUCTION

In the broadest sense, *size enlargement* can be defined as any process in which small particles are formed into larger entities. Size enlargement is used to improve the usefulness of fine materials either in a downstream processing step or as a final product agglomerate. As discussed in Section 1 below, there are many specific reasons for doing size enlargement depending on the product and/or industry under discussion.

Most often the starting material for size enlargement is fine particles and the product is agglomerates or aggregates in which the original particles can still be identified. This is not always the case, however, since granular free-flowing solids may result from the drying or cooling of concentrated slurries or melts in which amorphous or crystalline masses are formed from the feed particles. Such processes are included as size enlargement techniques in the present treatment. In addition, it is often considered that the products of size enlargement should be permanent masses and indeed methods to produce permanent bonding (e.g. heat induration of mineral agglomerates) are discussed in Chapter 6. This criterion is not entirely satisfactory in that the bonding necessary in a given application may be quite weak and transient; agglomerate strength need only be sufficient for the products to meet downstream requirements. Thus relatively weak powder clusters suffice for instantized food products and in the preparation of powders for tableting. It should also be noted that size enlargement may be only a secondary result in some processes where granular materials are produced. The primary objective may be, for example, drying (as in spray and flash drying) or disposal of waste (as in fluid bed incineration). A number of such peripheral methods are included here with emphasis, of course, on the size enlargement aspects.

1. Objectives of size enlargement [1–3]

Size enlargement processes are used in many industries today with the desired results depending on the particular application. In each case, however, the substitution of granular material for fine powders yields a number of beneficial effects. The benefits and objectives of size enlargement, together with some examples of their application, may be summarized as follows:

1. Production of *useful structural forms and shapes*, as in the pressing of intricate shapes in powder metallurgy or the manufacture of spheres by planetary rolling.

TABLE 1.1

Historical development of size enlargement methods

	Ceramics	Powder metallurgy	Medicinal and pharmaceutical
Early usage	Glassmaking, pottery forming, preparation of clay building materials in antiquity.	Ancient technique of hammering sponge iron into implements. Precious metal objects formed from powder.	Solid molded forms containing medicinal ingredients can be traced back at least 1000 years.
1800	Development of mechanized forming methods essentially the same as modern techniques.	Platinum powder compaction, followed by heating and hot forging to shape.	
1850			Development of medicinal tablets by die compaction of powders. Early single punch and rotary tablet machines in use. Design of both rotary and reciprocating tablet machines became well-defined.
1900		Industrial compaction, sintering and working of powders (especially tungsten) for incandescent filaments. Expansion of iron and copper parts from powder; related to mass production in automotive industry, WWII, etc.	Output rates of tablets increased. Improvements in physical and chemical characteristics of compressed tablets. Research and improvements in functional aspects (disintegration, dissolution).
1950	Development of non-silicate ceramics with electronic, nuclear and space applications.		
	Growth of materials science, composite materials.		

Coke, coal and other fuels	Minerals processing	Fertilizers	Chemical, food and other industries
Coal carbonization developed to yield coke (coal agglomerates) and byproducts.	Batch sintering to handle fines included roast heaps, blast roasting pots.		
Large scale production of pressed coal blocks from coal fines and pitch binders.	Iron ore briquetted and fired.		Molding of rubber and resinous compositions.
Binderless briquetting of lignite, peat, etc. Development of roll and extrusion presses for fuels.	Continuous sintering of fine ore and dusts.		
	Burden preparation in tumbling drums.	Early development of granular nitrogen fertilizers usually based on rotary dryer.	Hot molding of thermosetting plastics.
		Granulation of mixed fertilizers widely adopted in U.K. before 1950.	Pelleting of carbon black.
	First commercial iron ore pelletizing plants in operation.	After 1950, a major swing to granular mixed fertilizers in U.S.	
Development of formed coke technology, based on coal agglomerates.	Dramatic increases in plant capacity.		"Instant" agglomerated foods (e.g. milk powder) developed.

2. Provision of *definite quantity units* for metering, dispensing and administering, as in pharmaceutical tablets.
3. Reduction of *dusting losses*, as in the briquetting of waste fines.
4. Creation of *non-segregating blends* of particulates, as in the sintering of fines in the steel industry.
5. Improved *product appearance*, as in the manufacture of fuel briquets.
6. Reduced *caking and lump formation*, as in the granulation of fertilizers.
7. Improved *flow properties*, as in the granulation of ceramic clay for press feed.
8. Increased *bulk density* with improved storage and shipping properties, as in the pelleting of carbon black.
9. Decreased *handling hazards*, especially with irritating or obnoxious powders, as in the flaking of caustic.
10. Control of *solubility*, as in "instant" food products.
11. Control of *porosity* and *surface-to-volume ratio*, as in the pelleting of catalyst supports.
12. Improvement in *heat transfer*, as in the agglomeration of ores and glass batch for furnace feed.
13. *Separation of multicomponent mixtures*, as in selective agglomeration of the combustible matter in coal.
14. *Removal of particles from liquids*, as in the formation of pellet-like flocs from clay in water by the use of polymeric bridging agents.

2. Historical perspective [1,3–18]

Table 1.1 summarizes the major developments in size enlargement processes that have taken place over the past 200 years. Related techniques can be traced to forming processes used in antiquity, including the preparation of building materials such as bricks and tiles, the forming of solid objects from sponge metal by hammering and the administering of medicinal compounds in various solid forms. Agglomeration became established as a relatively large-scale practical operation during the industrialization of the 19th century with the need to beneficiate and process fine coals and ores. Size enlargement became a basic step in many industrial processes in the first part of this century and has enjoyed particularly rapid expansion in the last thirty years. Some of the factors that have contributed to this growth are:

- a. Intensive agriculture and the use of high analysis nitrogen fertilizers which cake badly in non-granular form.
- b. Reduction in the quality of resources and the necessity for grinding to liberate impurities followed by agglomeration of the upgraded material.
- c. Environmental factors, including the disposal of recovered dusts and the substitution of coarser furnace feeds to avoid airborne fines and fumes.

- d. The use of automated high-volume processing requiring consistent feeds with good flow properties.
- e. A modern trend to instant or convenience food products.

3. Classification of methods and scope of book

Methods to create larger entities from fine particles may be broadly classified into two main categories [1]. On the one hand are the forming-type processes in which the properties of the *individual agglomerates* (such as shape, size, composition, density, etc.) are carefully controlled. On the other hand are the size enlargement methods in which a coarser granular material is created from fine powders. The properties of the *bulk material* are controlled in this case and the characteristics of the individual agglomerates are important only insofar as they affect the properties of the whole or bulk product. As a result of these differences, the forming-type methods are usually of low capacity, often measured in pieces/hour, while the methods to beneficiate bulk material are of much larger capacity, usually measured in tons/hour.

As seen in Table 1.1, forming methods using fine powder feeds have been practised for centuries in the preparation of such products as tiles, bricks and tablets. Brief descriptions of modern forming methods used mainly in the pharmaceutical, powder metallurgy and ceramics fields are given in Table 1.2. Such forming methods are generally outside the scope of the present treatment. Information on these techniques is well documented elsewhere; the reader is directed to the references noted in Table 1.2.

Size enlargement to improve the bulk properties of particulates is the subject of this book. Techniques to accomplish this beneficiation may be classified according to the principal method used to bring particles together into agglomerates. The categories used here are:

1. Agitation methods — tumbling agglomeration
2. Agitation methods — mixer agglomeration
3. Pressure methods
4. Thermal methods
5. Spray and dispersion methods
6. Agglomeration from liquids

The methods available in each of these categories are briefly described in Table 1.3 together with an indication of the equipment used, capacity range, agglomerate characteristics and some of the advantages and limitations of each. Full details are given later in the book where a chapter is devoted to each major category of methods. Table 1.4 summarizes the areas of application for the various size enlargement methods.

Of course, in any classification system areas of overlap are unavoidable. Some size enlargement processes involve more than one agglomeration mechanism. For example, thermal after-treatment is quite common to harden

TABLE 1.2

Forming-type methods; properties of individual compacts are controlled

Method	Description	Other names	Equipment
<i>Agitation methods</i>			
Planetary rolling and/or shaking	1. Fine powders, held together by capillary forces of liquid binder, grow into spheres under rolling collisional forces. 2. Fine powders formed into plastic mass with aid of liquids and binders, extruded into pellets which are rolled into spheres.	Spheroidization, spherical agglomeration Pill making, spheronisation	Modified planetary mill, various shaking devices Automatic pill machines, "Marumerizer"
Vibratory compaction	Vibration is applied to a mass of powder in a container-mold so that the particles seek a state of closest packing. Part subsequently strengthened, e.g. by sintering.		
<i>Pressure methods *</i>			
Unidirectional compaction	1. Powders compressed within die cavity into cylindrical or other simple shapes by action of punch. 2. Powders compressed into more complex shapes with special dies and plungers.	Tableting, powder pressing, dry pressing, hot pressing Compression molding, powder pressing, dry pressing, damp pressing, hot pressing	Tablet machines, powder presses. Powder presses, molding presses
Isostatic compaction	Powder encased in flexible mold is pressed equally from all directions by pressure transmitted to the mold by a liquid medium.	Isostatic molding, isostatic pressing	Hydrostatic molding chambers, automatic isostatic presses
High energy rate compaction	Powders subjected to extremely high pressures instantaneously to form compacts.	High energy rate forming (HERF), explosive forming	High velocity presses, impact presses, explosive rams.
Roll pressing	Powders compacted by squeezing between two rolls.	Powder rolling, roll compaction	Roll presses
Extrusion	Stiff plastic particle mix is pushed through a die orifice to form a continuous compact which may be cut to desired length.		Vacuum auger, screw extruder, plunger press, piston extrusion apparatus

Capacity	Advantages, limitations, comments	References
Batch process generally using less than 0.5 kg per batch.	High energy input yields more uniform size, greater sphericity and density than other agitation methods. Less waste than pressing-sintering-grinding method. Sphere size generally 0.1 mm to 5 mm.	19, 20, 21
Batch process; can produce 200 to 2000 kg per hour.		9, 22
	Size distribution, shape, etc. of particles must be controlled for maximum density. May be used to obtain more uniform density distribution in complicated shapes.	23
Single-punch machines, up to 200 pieces/min; Rotary machines, up to 2000 pieces/min; High speed machine up to about 10,000 pieces/min.	Materials should have good flow characteristics and cohesive properties. Feed preparation, binder, lubricants used to meet requirements. Can produce compacts of simple shape with controlled properties at high rates.	9, 24, 25
In powder metallurgy, rate may range from 5 parts/min to 25 parts/min.	Non-uniform pressure distribution in compact limits aspect ratio of parts. Can be improved by pressing from top and bottom, rather than from one direction only. Tooling cost relatively high but powder pressing often more economical than other methods.	5, 7
About 1000 to 1500 pieces per hour with automatic presses.	Compacts with uniform properties (e.g. density, shrinkage) are formed. Wider dimensional tolerances than with unidirectional compaction are required. Complex and large shapes are possible. Lubricants not required.	26
	Can produce very large structural shapes. Used with powders requiring high pressures to produce compacts of special properties.	5
	High capacity strip-forming process.	27
	Particulates must be capable of forming a plastic mass with liquid. Plasticizers may be used to accomplish this. Compact shape must have an axis normal to a fixed cross section.	12, 28
	Dense compacts can be formed rapidly and economically at low pressures. Piston system operates at higher pressure than auger and can produce more precise compacts. Less precise, however, than closed die compaction.	

TABLE 1.2 (*continued*)

Method	Description	Other names	Equipment
<i>Thermal methods</i>			
Sintering	1. Strengthening of compacts by heat treatment through bonding of particles by molecular (or atomic) attraction in the solid state. Attendant processes include partial fusion, densification, recrystallization and/or chemical reaction.	Firing	Kilns, Furnaces
	2. Powdered polymer fused at inner surface of hot hollow mold, excess powder poured out and remainder completely fused in oven. Hollow article removed from mold after cooling.	Powder molding	Ovens
<i>Miscellaneous methods</i>			
Soft plastic forming	1. Sufficient water is added to clay body to form a soft plastic mass which is readily worked at low pressures.	Hand molding	Potter's wheel
		Ramming	Pneumatic tamping tools, mold.
		Jiggering	Revolving mold, profile tool.
	2. Suitable pharmaceutical formulations are mixed to a plastic mass using solvent and/or moistening agents, forced into a mold, pushed out and dried.	Tablet molding	Hand molding, automatic tablet molding machines
Forming from suspension	1. Stable suspension of particles is poured into porous mold (plaster of Paris) and solid particles are deposited as water is absorbed by mold.	Slip casting	
	2. Dispersion of finely divided poly (vinyl chloride) in liquid plasticizer poured into mold which is heated to 350–400° F to form fused solid on interior of mold.	Slush molding	

* May also include simultaneous heating to yield superior compacts of higher density, finer grain size, close dimensional tolerance, etc.

Capacity	Advantages, limitations, comments	References
	Common final treatment for ceramic and metallic compacts. Sintering treatment normally occurs below melting point of powder material.	5, 23
	Used to form large pipe sections and tanks from polyethylene.	7
	Not a precise forming method. Jiggering requires shapes with symmetrical circular cross sections.	12, 28
About 2500/minute for automatic equipment	Molded tablets are generally softer and more readily soluble than compressed tablets.	9
	Objects formed can be solid or hollow of controlled wall thickness. Suitable for complex shapes and large parts. Cost of molds is low.	12, 28

TABLE 1.3

Methods to improve properties of bulk powders by particle size enlargement

Method	Description	Other names	Equipment
<i>Agitation methods</i>			
Tumbling agglomeration	Powders, usually in the presence of a liquid binder, are subjected to a rolling cascading action to form agglomerate nuclei which grow by coalescence and/or layering of fines.	Granulation, balling, wet pelletization, pelletization, pelleting	Drums, inclined discs, cones, pans, bowl and plate granulators, "Flying Saucer"
Mixer agglomeration	Moist, plastic particles are mixed and "fluffed" to a nodular texture by the action of (usually) twinshaft agitators in a horizontal cylindrical vessel.	Granulation, wet pelletizing	Horizontal pan, pugmill, drum pugmill, blunger, peg granulator, pin mixer
Powder clustering	Powders are superficially moistened (often at less than 5% water or liquid binder using spray, steam, mist, etc.) during agitation to form fine agglomerates.	Granulation, instantizing	Powder blenders (conical, vertical shaft, ribbon), falling curtain agglomerators (drum, vibrating feeder), continuous flow jet mixing systems
<i>Pressure methods</i>			
Unidirectional, piston-type compaction	1. Large briquets formed (e.g. from scrap metal turnings) by piston compression into die.	Briquetting, compacting, preforming	Heavy duty compacting presses
	2. Vibration and limited ram pressure consolidate granular material in molds of various sizes and designs.	Briquetting, compacting, molding	Table presses, concrete block and brick machinery
	3. Powders compressed into flat-faced tablets by action of punch and die, comminuted and screened to form feed for final compression.	Dry granulation, slugging, pre-compression	Heavy duty tableting press
Roll pressing	1. Particulate material, with or without binder, is compacted by squeezing as it is carried into the gap between two rolls rotating at equal speed	Briquetting, roll compacting	Compacting rolls with smooth, corrugated or pocketed surfaces

Typical capacity per machine	Advantages, limitations, comments
Up to 100 tons/hr for iron ore balls; 200 tons/hr or more for sinter preparation; 50 tons/hr for fertilizer granulation.	Ball-shaped agglomerates only can be formed. Diameters from about 1/8 in. are normally produced. Larger balls require very fine feed (e.g. 80% minus 44 μ m) although much coarser feeds can be used for smaller product. Rather weak agglomerates are formed; must be further strengthened e.g. by drying or firing. Rotary drums and inclined discs are most common. Discs yield more uniformly-sized product and require less space. Drums are less sensitive to changes in feed and are more suited to simultaneous treatment of charge (e.g. drying, ammoniation) as in fertilizers.
Generally less than 10-15 tons/hr, but much larger units are in use (e.g. 350 tons/hr fertilizer blunger, 300 tons/hr drum pugmill for sinter preparation)	Positive "cutting-out" action allows plastic and sticky masses to be treated. Kneading action claimed to produce denser, stronger granules. Less plasticizing liquid phase required than with tumbling agglomeration. For plastic material, less power cost than extrusion. Irregular agglomerates may be formed, requiring finishing by tumbling, e.g. in dryer. Product size distribution wide, requires high recycle. With very fine powders, capable of forming micropellets e.g. finer than 10 mesh. Upper size usually less than 5-6 mm. Agitator wear must be considered.
Generally less than 10 tons/hr, but units up to about 50 tons/hr, also available.	Used to produce small (less than 2 mm), irregular, relatively weak agglomerates with fast dissolving and wetting properties. Generally not suitable for materials which go through a plastic or sticky stage. Blending equipment yields good uniformity. Jet-mizing systems require well-mixed feed but are capable of larger throughputs.
Less than 5 tons/hr	Briquets may be 5 in. dia. \times 3 in. thick. Simple operation. Not suited to large tonnages. Reciprocating nature involves non-uniform loads on drive motors.
50 tons/hr quoted for iron fines	Relatively larger agglomerates formed than in most other methods. Limited pressure necessitates binder for adequate final strength.
Related to tableting operation	"Slugs" or preforms are usually 1 to 2 in. diameter in pharmaceutical manufacture. Suited to granulation of moisture-sensitive materials and those unable to withstand high temperature of drying in wet granulation methods.
5 to 30 tons/hr for industrial chemicals, generally up to 50 tons/hr; some machines up to 100 tons/hr.	Probably most versatile tonnage method of size enlargement. Most materials can now be agglomerated with aid of binders, heat and/or very high pressures if needed. Many shapes are possible in compacts, but fine detail of tablets lacking. Flashing or web on compact may be objectionable. Sheet can be formed with smooth or corrugated rolls and subsequently broken down to granular product. Principal operating costs related to roll replacement (due to abrasion) and power consumption.

TABLE 1.3 (*continued*)

Method	Description	Other names	Equipment
Extrusion	2. Material is compacted as it is carried into the gap between a molding groove on the inner circumference of a ring and a press wheel of smaller diameter running in the groove.	As above	Ring roller presses
	1. Powders are intimately mixed to a plastic state and pushed through an orifice to form compact.	Pelleting, pelletizing, plasticizing, granulation	Piston extrusion presses, roll extrusion presses, pellet mills, screw extruders, plasticizing pans
	2. Granulation of moist mix by forcing it through a mesh screen or plate through action of wiper mechanism.	Granulation, sifting	Screen or sieve granulators
<i>Thermal methods</i>			
Sinter formation	Feed of ore fines, pulverized fuel, waste dusts and flux is moistened, mixed to a nodular texture and ignited on a horizontal grate to form an agglomerated sinter cake.	Sintering	Continuous sinter strand (travelling grate)
Pelletizing (cf. "tumbling agglomeration")	Green balls or pellets of ore or minerals are hardened by heat treatment with attendant loss of moisture and volatile matter.	Pellet induration, pellet firing	Travelling grate, rotary kiln, vertical shaft furnace
Nodulizing	Mixture of ore fines and fuel, or simply of fusible fines, is passed through a rotary kiln or dryer and agglomerates form by partial fusion, chemical reaction, etc. as the temperature is raised	Granulation, calcining	Rotary kiln, dryer
Drying and soldification	1. Plastic masses and pastes are preformed into wet agglomerates of consistent size by extrusion or granulation (cf. "pressure methods") prior to drying and hardening in various types of dryers.	Granulation, pelletizing, drying	Various pre-forming devices (extruders, granulators) followed by dryers
	2. Pastes and melts are spread as a thin film on a surface where they are dried and cooled, respectively. Subsequently scraped off to yield solid product.	Drum drying, flaking, melt cooling, endless belt processing, slating, pastille formation.	Drum dryers, drum flakers, endless belt systems

Typical capacity per machine	Advantages, limitations, comments
Generally less than 5 tons/hr	Press wheel may contain projections to form briquets from compressed strip. Pressure build-up and release more gradual than in roll press, claimed to avoid crack formation in agglomerates. Capacity of largest machines much less than for roll presses.
Up to 25-30 tons/hr or more	Cylindrical pellets formed by cutting with knife as extrudate leaves die. "Spaghetti" form also produced, as in wet granulation of pharmaceutical mixes by extrusion through orifice plate. Plastic mix formed by shearing, mixing action prior to extrusion. Binders, plasticizing agents, lubricants may be used. Relatively low pressure method particularly suited to sticky, cohesive materials.
Depends on mix consistency, screen mesh size and wiper speed, probably a few tons/hr.	Relatively low cost, simple granulation method.
Up to about 1000 tons/hr or more	Sinter is more irregular and cannot withstand handling as well as pellets. Hence, generally located at smelter where is well suited to utilization of waste materials. Can use relatively coarser feed than pelletizing.
Up to about 1000 tons/hr or more	Process developed to utilize very fine concentrates of low grade ores. Uniform size, spheroidal shape, abrasion resistance and strength well suited to long distance shipping from mine to smelter.
50 tons/hr	Formation of rings of fused material inside kiln is a problem. Technique can be used with fusible fertilizer formulations to granulate at low moisture levels. May not be suitable for materials which decompose on heating or if melting point too high.
Dependes on pre-forming method used.	Method simultaneously dries and granulates plastic masses such as pastes and filter cakes. Selection of best preforming method and dryer depends on experience and suitable tests.
A few 1000 lb/hr, up to 20 or 30 tons/hr.	A versatile system to handle a wide range of feed materials, capacities and product forms. Useful for heat-sensitive materials.

TABLE 1.3 (*continued*)

Method	Description	Other names	Equipment
<i>Spray and dispersion methods</i>			
Spray drying	Atomized liquid feed is brought into contact with a sufficient volume of hot air to evaporate liquid and solidify drops		Spray dryers
Prilling	Atomized liquid melt is cooled to solid agglomerates during fall through cooling medium	Spray cooling, solidification, congealing, shot formation	Spray chambers, prilling towers, shot towers
Spray onto dispersed powder (cf. powder clustering, agitation methods above)	Atomized liquid feed is sprayed into circulating dispersion of already-dried particles. Hot gases maintain dispersion.	Spray granulation, spouted and fluidized bed granulation	Fluidized and spouted beds, Wurster apparatus
Flash drying	Wet feed is dispersed and conveyed in high velocity stream of hot gas and dries almost instantaneously to granular product.	Pneumatic conveying drying	Flash dryers, conveying systems
Agglomeration in liquid media	Fine particles in liquid are agglomerated by addition of a bridging agent during agitation	Immiscible liquid wetting, selective agglomeration, spherical agglomeration, wet pelletization, pellet flocculation	Various mixers

Typical capacity per machine	Advantages, limitations, comments
Water evaporative capacities up to 40,000 lb/hr	<p>All spray methods are capable of producing powder directly from liquid (solution, slurry, emulsion, melt, gel, paste). Approximately spherical, homogeneous particles are formed with good flow properties. Elimination of other steps saves space, handling. Liquid must be pumpable and atomizable. Creation of large surface area gives large heat and mass transfer rates. Amenable to continuous, automated operation. Attrition and fines carryover in off-gas must be dealt with; added costs.</p> <p>Product size limited to 50 to 500 microns diameter; may require further size enlargement. Hollow particles often formed with low bulk density and/or fluffy character. May be disadvantage or advantage (e.g. quick dissolving). Few seconds residence time suited to heat sensitive, easily oxidized, explosive or flammable materials.</p>
Approx. 20 tons/hr for fertilizer materials.	<p>Suitable only for materials with sufficiently low melting point which do not decompose on fusion. Upper limit on prill size (about 3 mm) due to practical limit on tower height. Very tall towers only justifiable for large throughputs. Prilling into liquid requires shorter towers but extra step required to remove liquid medium.</p>
Up to about 50 ton/hr	<p>Longer residence time than spray drying, hence greater drying load (lower solids content of feed) can be maintained. Also, larger agglomerates (0.5 to 5 mm) can be produced. Spouted beds can produce larger agglomerates than fluidized beds. Any large differences between wet and dry material (e.g. particle size, density, stickiness) may preclude use. Less restriction on feed moisture content and more compact equipment than for prilling.</p>
Water evaporative capacities up to 20,000 lb/hr or more	<p>Very high thermal efficiency achieved. Less control of granular product size and size distribution than with other dispersion methods.</p>
Laboratory scale up to tons/hr, depending on application.	<p>Recovers particles directly from liquids. Can be selective in removing one or more particle types. Highly spherical agglomerates are possible.</p>

TABLE 1.4

Some applications of size enlargement methods.

	Minerals processing	Fertilizers	Powder metallurgy
<i>Agitation methods</i>			
Tumbling agglomeration (see also Table 3.1)	Agglomeration of ferrous and non-ferrous ores and dusts	Granulation of many types	
Mixer agglomeration	Sinter strand feed conditioning; pre-mixing for balling	As above	
Powder clustering			
<i>Pressure methods</i>			
Piston-type compaction		Tableting of some specialty items	Forming of many parts
Roll pressing (see also Tables 5.3, 5.5 and 5.6)	Many ores, coal, coke, waste fines, etc.	Mixed fertilizers, potash, urea, etc.	Continuous forming of sheet, strip and bar
Extrusion (see also Table 5.7)	Pelleting of fine concentrates, dusts, etc.	As above	Forming of sheet, bar stock, tubing, wire, etc.
<i>Thermal methods</i>			
Sintering	Agglomeration of ferrous and non-ferrous ores and dusts		Strengthening and densification of compacts
Pelletizing	Induration of balled ores		
Nodulizing	Iron ore agglomeration		
Drying and solidification (see also Table 6.10)	Formation of sulphur "slates"	Cooling and solidification of urea, ammonium nitrate	
<i>Spray and dispersion methods</i>			
Spraying drying	Mineral ore concentrates		Powder production

Ceramics forming	Pharmaceuticals	Food processing	Chemical and other industries
			Carbon black pelleting; many chemicals
Granulation of plastic clays; press feed preparation	Tablet feed preparation		Carbon black pelleting; microagglomeration of many products
	As above	Agglomerating instantizing (see Table 4.6)	Granulation of detergents and other chemicals
Forming of parts by soft "plastic" molding and dry pressing	Slugging (precompression), tableting		Plastic preforms, catalysts and supports, scrap metal recycling, industrial chemicals
	Dry granulation of tableting feed		Wide variety of applications (see Tables 5.5 and 5.6)
Pelleting clays, forming structural clay products (e.g. tile, bricks)	Wet granulation of tableting feed	Animal feeds, cereals, snack foods	Catalyst carriers, scrap plastics, many others (see Table 5.7)
Strengthening and densification of compacts			Plastic powder molding
			Formation of cement clinker
Drying of clay		Drying of liquid feeds to coarse powders	Cooling, flaking, drop-forming of resins, hot melt adhesives, resins, caustic, various chemicals
Press feed preparation	Tablet feed preparation	Rapidly dispersible powder products from liquids (see Table 4.6)	Plastics, resins, washing powders, dyestuffs, etc.

TABLE 1.4 (*continued*)

	Minerals processing	Fertilizers	Powder metallurgy
Prilling (see also Table 7.1)		Production of ammonium nitrate, urea	
Spouted and fluid bed systems		Granulation of ammonium nitrate, complex fertilizers	
Flash drying	Drying and granulation of clays, diatomaceous earths		
<i>Agglomeration in liquid media</i>			
	Fine coal preparation		Sphere formation

agglomerates after they are initially formed by some other method. Where such grey areas occur, cross-references will be given to related material.

4. Selection of size enlargement methods

A simple approach to specifying a suitable size enlargement method for a given application is by analogy to techniques currently used for similar materials. If a similar material can be identified in the applications summary of Table 1.4 (or in the more detailed information given in later chapters), and if process objectives are similar in terms of agglomerate size, strength, etc., then the methods used for the established product may well be applicable to the new material.

A more fundamental approach to process selection first requires a clear definition of the problem and size enlargement objectives. This is followed by comparison with the capabilities of the available processes as catalogued in Table 1.3 and in greater detail in later chapters. Promising methods can then be selected and the clearly unsuitable methods ruled out. Factors to be considered in this comparison include:

1. Feed characteristics. Is the material sufficiently fine to ball it by tumbling agglomeration? Is it sufficiently plastic to allow extrusion? If a slurry

Ceramics forming	Pharmaceuticals	Food processing	Chemical and other industries
	Granulation of monoglycerides, carbohydrates, active ingredients in wax		Granulation of waxes, sulphur, resins, caustic soda, etc.
Clay granulation for pressing	Tablet feed granulation	Drying and granulation of starches	Fluid bed waste incineration, sulfur granulation, calcination of nuclear reactor wastes
			Drying and granulation of by-products and wastes.
			Removing soot and oil from water; sludge dewatering.

or paste, can it be pumped and atomized as required in the spray methods? Is the material heat sensitive and does this rule out some thermal methods? Questions such as these specific to the application must be asked.

2. Capacity required. The number of available methods is reduced as the required production rate becomes larger.

3. Agglomerate size and size distribution. Certain methods, such as spray drying and powder clustering, yield only small agglomerates while others, such as briquetting, can yield very large agglomerates.

4. Agglomerate shape. Mixer agglomeration, fluid bed granulation and sintering yield quite irregular agglomerates. Balling produces only spherical shapes while extrudates are cylindrical in shape. Possible effects of different shapes on downstream utilization should be evaluated.

5. Agglomerate strength. Relatively weak products suited to some applications result from methods such as powder clustering and spray granulation. If very strong agglomerates are required, thermal hardening, pressure compaction or use of a suitable binder is indicated.

6. Agglomerate porosity and density. This is closely related to product strength. Pressure methods are well suited to control of porosity which may be needed in some applications.

7. Wet versus dry methods. Dry methods involve dust and may not be suitable if noxious chemicals and other dangerous materials are being treated. On the other hand, wet methods require after-drying with possible loss of

costly solvents. Some materials (such as pharmaceuticals) may be sensitive to wetting while others may recrystallize in a different form on drying.

8. Simultaneous processing possible? Certain methods and equipment, such as drum agglomerators, are suited to simultaneous processing such as chemical reaction.

9. Space limitations. For example, pressure compaction methods can give high throughputs from a relatively small installation while other methods, such as prilling, require a large vertical space for tower installation.

At this stage, a tentative selection of at least two alternative size enlargement methods can be made. These initial selections can then be refined with the help of laboratory and/or pilot plant tests. Most vendors (see Appendix, p. 177) have pilot equipment available on a rental basis and are prepared to assist with test runs and technical advice. A final process selection can then be made taking into account the normal considerations of reliability, flexibility, ease of maintenance and minimum overall cost at the required throughput.

5. Literature of size enlargement

Although size enlargement is an established operation in many industries, information on the subject has not always been readily available. This is partly due to fragmentation of available data, with articles published in journals specific to the various areas of application. The references given with each chapter of this book constitute a reasonably comprehensive library of information, at least in the English language. In addition to these specialized articles, a number of books and reviews are now available which give a more general treatment of the unit operation of Size Enlargement. For the convenience of the interested reader, these works have been collected in the references at the end of this chapter. Recommended sources include the Proceedings, Vol. 1 to 15, of the Institute for Briquetting and Agglomeration (see references 14 and 29, for example) and references 1, 2, 4–9, 11, 12, 15, 24, 26, 27, 30–40.

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AGGLOMERATE BONDING

1. Bonding mechanisms

The forces of cohesion between small particles are difficult to measure directly due to their very low magnitude and limitations resulting from small size. Nevertheless, a good understanding of the interaction between particles in an assembly is fundamental to size enlargement processes. For example, during the formation of agglomerates by agitation methods, relative bond strength determines growth mechanism and kinetics and influences agglomerate shape. In addition, bonding is important in determining the final properties of the product such as the ability to withstand handling during further processing, its rate of dissolution or reactivity, its density, etc.

In considering agglomerate strength, classifications directly related to the specific processing stages in an industry are traditionally used. Thus, in iron ore agglomeration, wet bonding, dry bonding and fired bonding are all important in the process sequence and each step has specific requirements and problems to be dealt with. A more fundamental approach, based on the nature of the particle-particle interaction and independent of the process step producing the interaction, was introduced by Rumpf and co-workers [1]. This classification of bonding mechanisms has become widely accepted in the literature and, together with Rumpf's theoretical model to estimate agglomerate strength, will be adopted here. The Rumpf classification is summarized in Table 2.1 together with some representative examples of the occurrence of the bridging mechanisms. In practice, more than one bonding mechanism may be acting simultaneously. Thus, in bonding by tar deposited by solvent evaporation, it is likely that oxidative hardening will also occur. In sintering ores, it is likely that bonding through chemical reaction will also contribute to strength. With very fine powders, it is difficult to determine whether bonding through long-range forces or adsorption predominates. Although mechanical interlocking of particles influences agglomerate strength, its contribution is generally considered to be small in comparison to other mechanisms.

Information on the cohesion of particles is obtained from theoretical considerations or from direct measurements on large particles or on powder masses and agglomerates. In this chapter, theoretical approaches are first considered, followed by an account of experimental methods and results.

TABLE 2.1

Classification of binding mechanisms according to Rumpf [1].

Class	Mechanism	Representative examples	References
1. Solid bridges	1. Sintering, heat hardening	1. Induration of iron ore pellets	31
		2. Sintering of compacts in powder metallurgy	57
	2. Chemical reaction, hardening binders, "curing"	1. Cement binder for flue dust pellets	58
		2. Ammoniation/granulation of mixed fertilizers	59
		3. Oxidation of tar binders	60
	3. Incipient melting due to pressure, friction	1. Briquetting of metals, plastics	43,61
	4. Deposition through drying	1. Crystallization of salts in fertilizer granulation	56
		2. Deposition of colloidal bentonite in dry iron ore balls	31
2. Immobile liquids	1. Viscous binders, adhesives	1. Sugars, glues, gums in pharmaceutical tablets	46
	2. Adsorption layers	1. Instantizing food powders by steam condensation	62
		2. Humidity effects in flow of fine powders	
3. Mobile liquids	1. Liquid bridges (pendular state)	1. Flocculation of fine particles in liquid suspension by immiscible liquid wetting	63
		2. Moistening/mixing of iron ore sinter mix	31
	2. Void space filled or partly-filled with liquid (capillary and funicular states)	1. Baling (wet pelletization) of ores	31
		2. Soft plastic forming of ceramic powders	64
4. Intermolecular and long-range forces	1. Van der Waals forces	1. Adhesion of fine powders during storage, flow and handling	15
	2. Electrostatic forces 3. Magnetic forces	2. Spontaneous dry pelletization of fine powders (e.g. carbon black, zinc oxide)	65,66
5. Mechanical interlocking	1. Shape-related bonding	1. Fracturing and deformation of particles under pressure	67
		2. Fibrous particles, e.g. peat moss	68

2. Theoretical tensile strength of agglomerates

Distinction must be made between systems in which bonds are localized at the points of particle contact and those in which the void space between particles is partially or completely filled with strength-transferring substance. Localized bonding is considered first, while some binder-filled systems are treated in Section 2.4.

2.1. Particle assembly with localized bonding

The mean tensile strength of an agglomerate can be estimated from a model [1] based on Fig. 2.1 and the following assumptions:

- (1) a large number of bonds exists in the stressed cross section,
- (2) a statistical distribution of bonds exists over the fracture section and over the directions in space,
- (3) the particles consist of a large number of monosized spheres which are statistically distributed in the agglomerate,
- (4) the bond strength between individual particles can be replaced by a mean value applicable throughout the whole assembly.

Statistical-geometrical considerations yield [1] the following equation:

$$\sigma_T = \frac{9}{8} \frac{1-\epsilon}{\pi d^2} kH \quad (1)$$

in which σ_T is the mean tensile strength per unit section area, ϵ is the void fraction in the assembly, d is the diameter of the spherical particles, k is the mean coordination number (average number of points of contact between one sphere and its neighbours), H is the tensile strength of a single bond.

Equation (1) indicates the influence of the major parameters which determine the tensile strength of an agglomerate with bonding localized at the points of contact. It requires knowledge of the tensile strength of a single

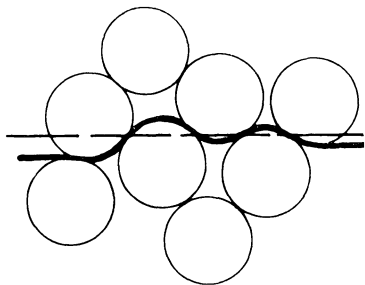


Fig. 2.1. Schematic representation of the ideal (— — —) and real (————) fracture area through an agglomerate [1].

bond and of ϵ , d and k . Void fraction, ϵ , is obtained from the density of the packing, ρ_b , and of the solid particles ρ_s :

$$\epsilon = 1 - \frac{\rho_b}{\rho_s} \quad (2)$$

Particle diameter, d , is known from size analysis.

The coordination number, k , presents a greater problem. Although k is strictly a function not only of the void fraction but also of the packing arrangement [2], experimentally determined coordination numbers may be correlated directly with porosity as an approximation. Thus, Rumpf [1] used the expression

$$k\epsilon \approx \pi \quad (3)$$

which, when substituted into eqn. (1), leads to:

$$\sigma_T \cong \frac{9}{8} \frac{1 - \epsilon}{\epsilon} \frac{H}{d^2} \quad (4)$$

2.2. Particle-particle bonds

In practice, the value of H in eqn. (4) cannot be calculated from theory for many of the interparticle adhesive mechanisms listed in Table 2.1. The weaker bonding mechanisms due to van der Waals forces, electrostatic attraction and mobile liquid bridges can be computed for simple model geometries as is discussed below. Unfortunately, the stronger classes of bonding due to the many forms of solid bridging and high viscosity liquids are amenable to theoretical treatment only in the simplest of cases. For example, if it is assumed that a solid bridging material with constant tensile strength, σ_s , is distributed over all particle-particle contacts and that the assembly fails through these bridges only, eqn. (5) results for the strength of the assembly [3].

$$\sigma_T = \frac{M_s}{M_p} \frac{\rho_p}{\rho_s} (1 - \epsilon) \sigma_s \quad (5)$$

where M_s/M_p is the weight ratio of binding material to particles, ρ_p and ρ_s are the densities of the particles and binding material, respectively, and ϵ is the void ratio of the assembly.

The complexity involved in those cases in which an interparticle bonding material is present can be appreciated from an analogy used [4] to describe an adhesive-bonded joint as a chain of at least five links. In the case of particle-particle bonding, these links include the cohesive strength of one particle, the interfacial bond strength of the bonding material to this particle, the cohesive strength of the bonding material itself, the interfacial bond strength to the second particle and finally the cohesive strength of the

second particle. The actual strength of the bond is essentially the strength of the weakest link. Thus, although theoretical estimates of the primary (short-range chemical) bonds and of the secondary (long-range van der Waals, electrostatic) bonds involved in this chain may be available [5], these estimates are of little value due to the presence of other effects which determine the strength of the total bridge. These effects may be due to, inter alia, the elastic response and extent of surface asperities on the particles, the existence of flaws or residual stresses in the binder or at the interface and the presence of surface impurities such as oxides.

2.3. Intermolecular and long-range bonds

In the absence of interparticle bonding material, the forces of adhesion are reasonably well understood and are orders of magnitude weaker than those discussed above. The results of many theoretical and experimental investigations of these long-range forces have been summarized by Krupp [6].

For very fine particles with intimate surface contact, these relatively weak secondary adhesive forces can be quite significant. For example, Rumpf [1] has estimated the contributions of van der Waals and electrostatic forces in agglomerates of fine-grained material. The calculations were made for quartz glass and yielded a binding force due to *van der Waals forces* between two spheres given by:

$$H = 4.2 \times 10^{-14} \frac{d}{a^2} \frac{\text{dynes}}{\text{cm}^2} \quad (6)$$

where, as shown in Fig. 2.2, d is the diameter of the spheres and a the separation of their surfaces, both in cm.

For an agglomerate of medium porosity ($\epsilon = 0.35$), eqn. (4) yields a tensile strength given by:

$$\sigma_T = 8.4 \times 10^{-20} \frac{1}{a^2 d} \frac{\text{kg}}{\text{cm}^2} \quad (7)$$

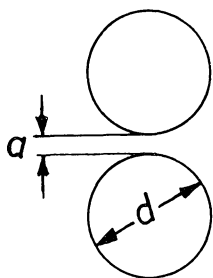


Fig. 2.2. Definition of symbols used in eqn. (6) for calculation of van der Waals attraction between two particles [1].

For an agglomerate of $1\text{ }\mu\text{m}$ diameter spheres separated by a distance of $30\text{ }\text{\AA}$, a tensile strength of 10 g/cm^2 (0.1 lb/in.^2) results from eqn. (7).

From approximate calculations, Rumpf concluded that *electrostatic forces* have negligible influence on the strength of agglomerates. Goldstick [7] estimated that the maximum interparticle force due to magnetic attraction may be many times larger than the maximum strength due to electrostatic forces but is nevertheless small, even in comparison with van der Waals forces.

2.4. Mobile liquid bonding

The various regimes of low-viscosity liquid which can exist in an agglomerate are depicted in Fig. 2.3. For regular systems of spherical packing, the cohesive forces have been calculated [1,8–10]. These forces originate with the interfacial tension at the liquid surface and the pressure deficiency (suction) created within the liquid phase by curvature at the liquid surface.

At low liquid levels, discrete lens-shaped rings are formed at the points of contact of the particles (Fig. 2.4). This is the *pendular state* of liquid content which persists until the liquid rings begin to coalesce. For uniform spheres, this occurs when:

for *cubic packing*, $\theta = 45^\circ$, % pore volume occupied by liquid = 18.2;

for *rhombohedral packing*, $\theta = 30^\circ$, % pore volume occupied by liquid = 24.3.

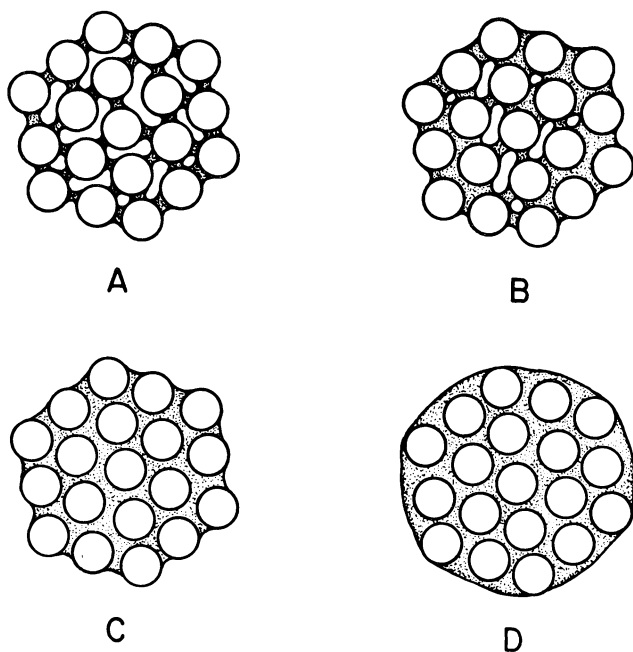


Fig. 2.3. Mobile liquid bonding; states of liquid content in an agglomerate. (A) pendular state; (B) funicular state; (C) capillary state; (D) particles in liquid droplet.

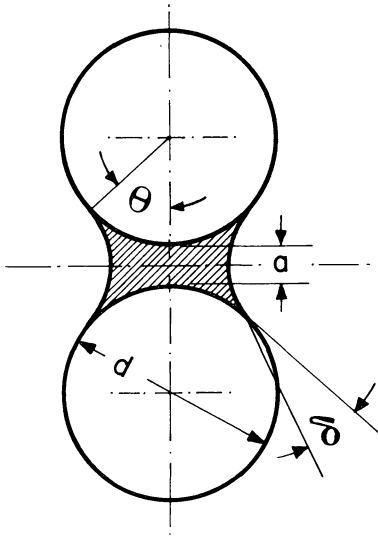


Fig. 2.4. Definition of symbols used in eqn. (8) for calculation of cohesive force between two spherical particles with a liquid bridge [13].

In the pendular state, bonding is localized at the points of particle contact and calculated values of the cohesive force between two particles may be substituted directly for H into eqn. (4) to yield the tensile strength of the assembly. For two particles in contact, H is given by [8,10]:

$$H = \gamma d \left(\frac{\pi}{1 + \tan \frac{1}{2}\theta} \right) = \gamma d F(\theta) \quad (8)$$

where γ is the surface tension of the liquid, d is the particle diameter, and, in Fig. 2.4, θ is the semi-angle of the liquid ring, $\delta = 0$, that is, the liquid completely wets the solid.

The function $F(\theta)$ is shown in Fig. 2.5 (curve with $a/d = 0$) plotted as a function of the percentage saturation, S , the percentage of total pore volume occupied by liquid. Two different curves are shown for $\epsilon = 0.35$ and $\epsilon = 0.45$ since for a given value of θ in Fig. 2.4, S assumes different values dependent upon the total pore volume of the assembly. Note that, in theory, the tensile strength of an assembly in the pendular regime rises steadily as moisture is removed since a very high pressure deficiency is predicted in the liquid bridge as it recedes and surface curvature increases. In practice, the tensile strength is found [11,12] to hold approximately constant or diminish with decreasing moisture content in the pendular state. This discrepancy has been explained [13,14] by the fact that no absolutely smooth particles are found

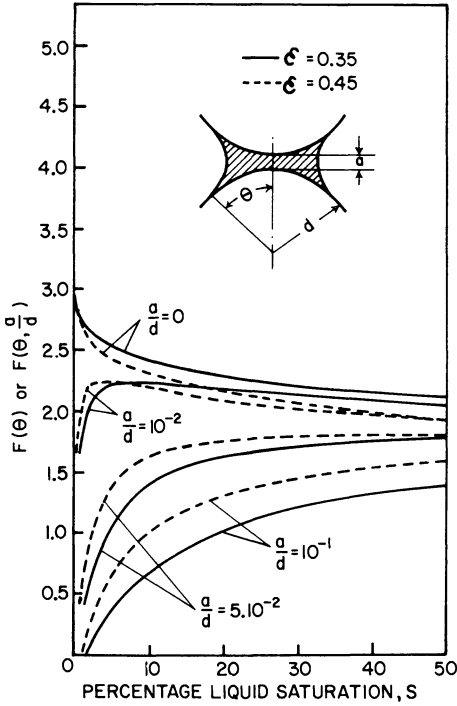


Fig. 2.5. $F(\theta)$ in eqn. (8) and $F(\theta, a/d)$ in eqn. (9) as a function of liquid saturation [13].

in practice and hence a separation “ a ” shown in Fig. 2.4 must be introduced to account for surface asperities. Then, eqn. (8) becomes:

$$H = \gamma d F\left(\theta, \frac{a}{d}\right) \quad (9)$$

where $F(\theta, a/d)$ is a complicated function of θ and a/d whose approximate values are shown in Fig. 2.5 for $\delta = 0^\circ$.

It will be noted, for constant γ and d , that the tensile strength of a pendular state assembly in Fig. 2.5 remains approximately constant or decreases with decreasing moisture content, as the parameter a/d increases. Pietsch [15] suggests that $F(\theta, a/d)$ may be given the mean value of 2 throughout this region. When this value is used in eqn. (9) which is then combined with eqn. (4), the tensile strength of a pendular state agglomerate results for $\delta = 0^\circ$:

$$\sigma_T = \frac{9}{4} \frac{1 - \epsilon}{\epsilon} \frac{\gamma}{d} \quad (10)$$

As the liquid content of an assembly is increased beyond the point of ring

coalescence (Fig. 2.3B) the funicular state is reached with its continuous network of liquid interspersed with air. When all the pores become filled with liquid, the capillary state prevails (Fig. 2.3C).

In the *capillary state* the contribution to the tensile strength of a particle assembly due to the pressure deficiency in the liquid is much larger than that due to the interfacial tension at the surface of the assembly. The tensile strength of a capillary state agglomerate can therefore be equated to this pressure deficiency which can be calculated from the Laplace equation for a circular capillary:

$$P = \frac{2\gamma}{r} \cos \delta \quad (11)$$

where P is the pressure deficiency, and r is the radius of the capillary.

The capillary radius may be related [1,16] to the properties of the particle assembly by means of an hydraulic radius based on the specific surface of the particles and the void fraction of the assembly. Equation (12) results for an assembly of *uniform spheres*:

$$\sigma_T \cong P_e \cong 6 \frac{1-\epsilon}{\epsilon} \frac{\gamma}{d} \cos \delta \quad (12)$$

where P_e is the maximum or "entry" suction pressure.

For an agglomerate composed of *irregular particles* in which the liquid

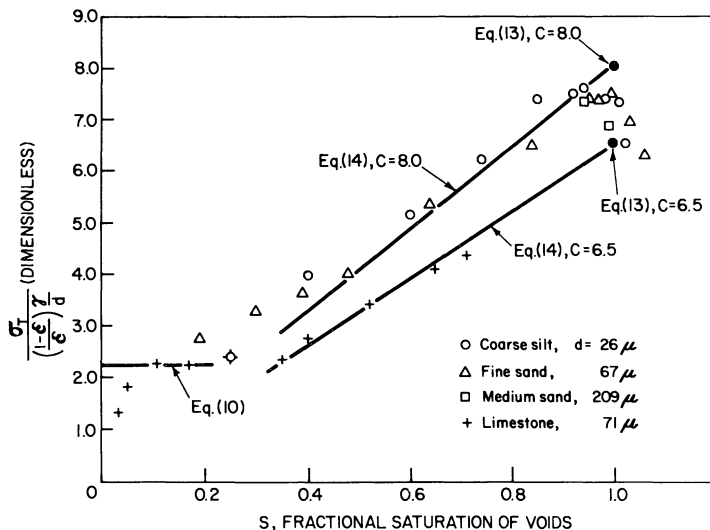


Fig. 2.6. Comparison of strength of moist agglomerates with predictions from capillary theory (data of Newitt and Conway-Jones [10] from compression testing and of Pietsch et al. [11] from tensile testing).

completely wets the solid, eqn. (13) may be written:

$$\sigma_T = C \frac{1 - \epsilon}{\epsilon} \frac{\gamma}{d} \quad (13)$$

where the constant C is related to the specific surface of the powder and accounts, inter alia, for the nonspherical shape of the grains. For sand particles, values of C ranging from 6.5 to 8 have been reported [10,16].

Comparing eqn. (10) and eqn. (13), tensile strength in the pendular state is about $\frac{1}{3}$ of that in the capillary state, while the funicular state yields intermediate values. As shown in Fig. 2.6, tensile strength in the funicular state can be approximated by:

$$\sigma_T = sC \frac{1 - \epsilon}{\epsilon} \frac{\gamma}{d} \quad (14)$$

where s is the fractional saturation of the voids. This equation is most accurate for moisture contents close to the capillary state.

With liquid contents *beyond the capillary state*, liquid completely envelops the particles (Fig. 2.3D). Only the interfacial tension of the convex surface of a continuous liquid drop tends to hold the particles captive.

3. Strength testing

Destructive test methods and equipment used to assess the quality of bonding in agglomerates or in powder compacts can conveniently be considered in two categories. The first group includes methods most suited to the research laboratory and provides information on the fundamental nature of the bonding. In this group are direct measurements of tensile strength and certain impression tests, especially on brittle compacts, which allow tensile strength to be calculated because the mode of failure is well understood. The second group of methods includes compression, impact, abrasion, attrition, dissolution, etc., methods. Although the results of this second group of tests are usually not well understood from a fundamental point of view, they are very important and more widely used than the first group because of their speed and simplicity, their relation to the subsequent handling and processing of the agglomerates and, often, their wide acceptance within an industry.

3.1. Tensile testing

Tensile (and compression) tests on agglomerates can be accurately and reproducibly performed with a variety of general purpose or specialty testing machines (see references 17 and 18, for example). These machines contain at least three elements for (1) gripping the specimen, (2) deforming it and (3) measuring the load required in performing the deformation. Other features

often provided include a measurement of deformation and a device to indicate the maximum load at which the specimen fails. In more elaborate testers, loading is done automatically by a motor at a variety of speeds, load is detected electronically with load cells employing strain gauges and results are presented on a strip chart recorder as a plot of load versus displacement.

In applying a tensile test to an agglomerate, the main problem to be overcome is to mount and grip the specimen without changing its properties during the procedure. Rumpf [1] used such a procedure in which a plane surface was first cut in the round pellet. This surface was then provided with concentric grooves. The cross section was coated with lacquer to provide a surface which was impenetrable by the adhesive subsequently used to fasten a metal adaptor. The pellet was then turned down in a lathe to a cylindrical shape of 18 mm (0.71 in.) diameter and a second plane surface parallel to the first was cut. After lacquering, a second metal adaptor was attached by adhesive. The finished specimen was finally mounted in a tensile test machine through the adaptors. During their preparation, the moist pellets were prevented from drying by storage in a saturated atmosphere.

An inherent weakness in this test procedure is the great care needed to avoid disruption of the specimen during cutting, mounting, and handling in general. The testing of dried pellets would obviously be much simpler. Many size enlargement operations, however, produce agglomerates with an outer shell of greater strength than the interior. Machining the specimen to a cylindrical shape interferes with this shell and the resultant reduction in overall strength must be taken into account [3] in Rumpf's procedure.

In a later publication [11], Pietsch et al. described a new apparatus designed to overcome the weaknesses of the earlier tensile testing procedure. Agglomerates were formed from powder with the desired moisture level in a ring-shaped cavity by compaction at about 70 kg/cm^2 (990 lb/in.^2). The agglomerate while still in the die was pulled apart to measure its tensile strength.

Ashton et al. [19] have designed an apparatus suitable for measuring the tensile strength of loosely coherent assemblies of particles. Although not suited to measuring the tensile strength of individual agglomerates, this tester can be used to determine powder strength in beds of particles held together by relatively weak bonds, for example those due to intermolecular forces [20] and low viscosity liquid bridges [21]. In this apparatus, the powder sample is consolidated into a shallow cylindrical cell which is split diametrically. One half of the cell rests on steel ball bearings and is movable; the other half is fixed. A horizontal load is applied to the movable half by means of a calibrated spring system. The force per unit area required to split the particle assembly can be calculated from the applied load and the fracture area.

Tensile strength can also be measured indirectly by means of a number of compression, bending and indentation tests in which, for brittle isotropic

materials at least, the principal internal stresses and failure mode are well understood. A number of such tests have been discussed by Berenbaum and Brodie [22]. The diametral compression test is one form of indirect tension test which has been applied to pharmaceutical tablets [23]. In this test, a thin wafer of compact is compressed diametrically between two flat platens. There exists across the loaded diameter a tensile principal stress of constant magnitude which tends to pull the wafer in half along this line. The magnitude of the stress is given by:

$$\sigma = \frac{2P}{\pi Dt} \quad (15)$$

where σ is the tensile stress, P is the applied load, D is the wafer diameter, and t is the thickness of the specimen. The tensile strength can be calculated from the breaking load by eqn. (15), provided failure in tension is observed [24].

3.2. Compressive testing

Compressive strength is widely used as an index of agglomerate quality. In the balling of iron ores, operators may perform this test for quality control with a hand-operated device which loads single pellets to fracture, but the result is very much operator-dependent [25]. In the laboratory, a compressive strength test can consist of crushing the agglomerate between flat parallel plates in which the lower plate is the pan of a balance which registers the load at failure [26]. In another variation [27], the lower plate was the pan of an equilibrated overhead beam balance. The agglomerate was loaded by allowing water to run from a graduated buret into a beaker placed on the other balance pan. The buret reading yielded the weight of water needed to cause failure. A number of compression testers are available which are dedicated to a particular industry although there is generally no reason to prevent their use in other areas. Stevenson and Bergstrom [25] have described such an instrument for use in iron ore pelletization while Brooke and Marshall [28] have compared the performance of four types of commercially-available "crushing-strength" instruments used in pharmaceutical tablet evaluation. In this latter study, the results showed inter alia, that varying methods of load application lead to variations in crushing strength values.

To obtain reproducible and accurate results, rate of loading and method of load application must be strictly controlled. This is especially true with plastic pellets, for example, the wet agglomerates produced in iron ore balling. Cahn and Karpinski [29] have shown that the fracture load for pliable agglomerates is highly dependent on the rate of loading. As shown in Table 2.2, a six-fold increase in deformation rate doubled the measured compressive strength of wet iron ore pellets containing bentonite which provides plasticity. After drying, these same pellets become much more brittle and an

TABLE 2.2

Effect of deformation rate on compressive strength of pellets [29] (wet iron ore agglomerates containing bentonite).

Deformation rate (in./min)	Fracture load (lb)	Average initial diameter (in.)
0.63	1.89 ± 0.34	0.56
1.15	2.75 ± 0.71	0.55
1.77	2.75 ± 0.84	0.56
2.68	3.25 ± 0.68	0.57
3.75	3.69 ± 0.66	0.57

increase in deformation rate by a factor of 23 increased the fracture load by only 20%. The low sensitivity to deformation rate of the measured compressive strength of brittle agglomerates is substantiated by the work of Kapur and Fuerstenau [30] with dry limestone agglomerates.

Two modes of operation are possible in applying a load to an agglomerate. With an automated tester the more common procedure is to move the platens at a constant rate; this is a constant deformation rate tester (CDRT). A second mode is possible in which the compressive force on the agglomerate is increased at a constant rate; this is a constant loading rate tester (CLRT). Cahn and Karpinski [29] provide an interesting comparison of these two modes of operation and conclude that the CLRT should be used when accuracy and precision are emphasized whereas the CDRT will suffice for routine testing.

3.3. Other tests of agglomerate strength

A number of other agglomerate strength tests, generally of the attrition/abrasion and impact varieties and of particular significance to a specific industry, will now be summarized.

In the *agglomeration of iron ores* [31,32], a drop test on the wet balls and abrasion/tumbling tests on the dried balls and fired (cold) pellets are routinely done in addition to compressive tests for quality control. The *drop test* is designed to indicate the ability of the wet agglomerates to withstand handling up to the point of their deposit on the drying and firing machine. Typically the balls are dropped from heights of 12 in. (300 mm) and 18 in. (450 mm) and the average number of drops prior to breaking yields the drop number. The *abrasion resistance* of dry iron ore balls was measured by Fitton et al. [33] by tumbling 500 g (1.1 lb) of balls in a 108 mm (4.3 in.) diameter cylinder 159 mm (6.3 in.) in length, constructed from 1.7 mm wire mesh, for 100 revolutions at one revolution per second. The amount of material passing through the screen as a fraction of the initial weight of balls

yields the abrasion index. In the ASTM *tumbler test* for iron ore pellets (E 279), an 11.3 kg (25 lb) sample in the size range 6.4 to 38.1 mm ($\frac{1}{4}$ to $1\frac{1}{2}$ in.) is placed in a tumbler drum 914 mm (36 in.) diameter by 457 mm (18 in.) long and rotated at 24 rpm for a total of 200 revolutions. The drum is equipped with two equally-spaced lifters 51 mm (2 in.) high. The abrasion index is given by the weight percentage of plus 6.3 mm ($\frac{1}{4}$ in.) material surviving the test and the dust index by the yield of minus 0.6 mm material. Other strength tests, such as the Linder rotating furnace procedure [34], are employed with iron ore pellets in an attempt to determine their reducibility and breakdown under the reducing conditions of the blast furnace.

In the *fertilizer industry*, a major reason for the development of granular products was the reduced interparticle contact between larger entities with consequent reduction in the *caking tendency* of the bulk product. If granule strength is too low, flow properties may be impeded and caking increased by granule degradation. Laboratory tests to assess caking tendency have thus been developed and consist of two parts [35]. The first part entails cake formation in a compression chamber under controlled conditions of air flow, humidity, temperature, etc. In the second part, the cake is removed and its crushing strength determined as a measure of the degree of caking.

In the *pharmaceutical industry*, tablets are examined not only for crushing strength [28] but also for dissolution rate [36]. This latter property is important in determining physiological availability of the active ingredient. In the *tablet disintegration test* outlined in U.S.P. XVII [37], tablets are contained in a basket-rack assembly which is immersed in a suitable dissolution fluid in a container. Agitation is provided by raising and lowering the tablets contained in the basket at a constant rate of 28 to 32 cycles per minute for a specified period of time. Acceptable tablets disintegrate completely by the end of the test period.

4. Experimental aspects of agglomerate bonding

4.1. Tensile and compressive strengths

As has been noted already, compression strength tests are widely used because of their speed and simplicity. Therefore, the relationship between this compressive strength most often used in practice and the theoretical tensile strength discussed in Section 2 will now be considered.

For the most common agglomerate shape, that is for ball-shaped agglomerates, compression strength is calculated as follows:

$$\sigma_c = \frac{L}{\frac{1}{4}\pi D^2} \quad (16)$$

where L is the compression force at failure, and D is the diameter of the agglomerate.

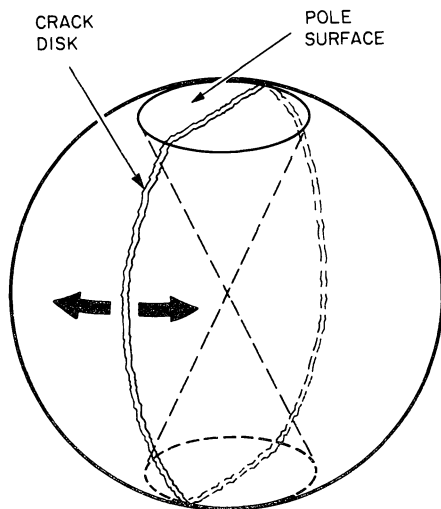


Fig. 2.7. Schematic representation of an agglomerate at fracture [30].

The relationship of the compression strength, σ_c , to the tensile strength, σ_T , can be appreciated from consideration of the mode of failure of a spherical pellet. During compression flat areas are produced (Fig. 2.7) at the poles where the load is applied. High local stresses acting on the interparticle bonds cause the particles to shear apart and to be driven into adjoining voids. Small, dense, wedgelike elements are thus formed in the regions adjacent to the pole surfaces and at failure the agglomerate splits in tension into two hemispherical sections along a circumferential crack joining the two poles of loading. This is the familiar “double-cone” failure mechanism. In translating the compressive load into a tensile stress through the formation of the double cone compacted areas, internal frictional effects between the moving particles must be overcome in addition to the tensile rupturing of the bonds in the crack plane. It follows that the compressive load at failure should be greater than the corresponding tensile value. For example, Rumpf [1] found for corresponding tensile and compression measurements on wet limestone pellets in the range $\sigma_T = 3$ to 12 lb/in.^2 (0.2 to 0.85 kg/cm^2):

$$\frac{\sigma_T}{\sigma_c} \approx 0.5 \text{ to } 0.77 \quad (17)$$

These values of σ_T/σ_c are shown in Fig. 2.8 as a function of the tensile strength, σ_T .

Newitt and Conway-Jones [10] noted that a rigorous theoretical derivation of the value of σ_T/σ_c requires the solution of equations of plastic deformation under conditions of axial symmetry for a granular material exhibiting

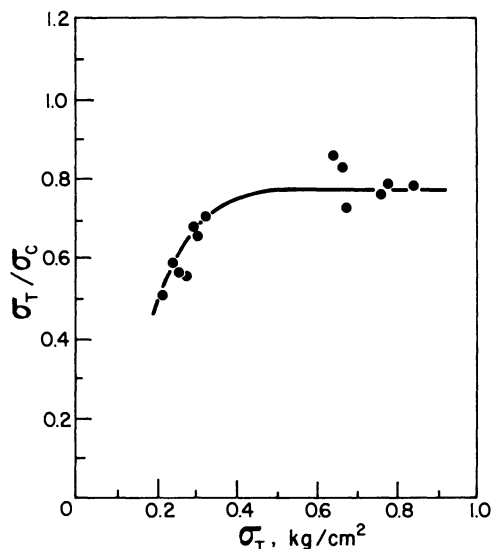


Fig. 2.8. Relationship between tensile and compression strengths of agglomerates [1].

internal friction. Orr [38] states that because of the internal friction present in compression tests, a linear relation between σ_T and σ_c is not expected but, as both strengths become greater, σ_T/σ_c should approach unity more closely. This agrees with Rumpf's results in Fig. 2.8.

4.2. Typical agglomerate strengths

The wide spectrum of compressive strengths encountered in practice is indicated in Fig. 2.9. The major variable determining these values is the particle-particle bonding mechanism. The increase in strength found as the bonding changes from the adhesion of submicron powders through capillary liquid bonding to the various forms of solid bridging is evident. This same trend is also shown in Fig. 2.10 which can be used to estimate order-of-magnitude values of agglomerate tensile strength as a function not only of bonding mechanism but also of particle size. This representation was originally conceived by Rumpf [1] based mainly on theoretical calculations. The inclined solid lines show the limits of tensile strength produced by the medium strength capillary and interfacial forces. The region below this refers to the relatively weak intermolecular and long range forces and forces due to adsorbed liquid films. The third region consists of the much stronger bonding due to the various mechanisms of solid bridging. As Rumpf has noted, the bonding strengths in this upper region are less dependent on particle size. For example, in briquetting, the active bonding surface depends strongly on

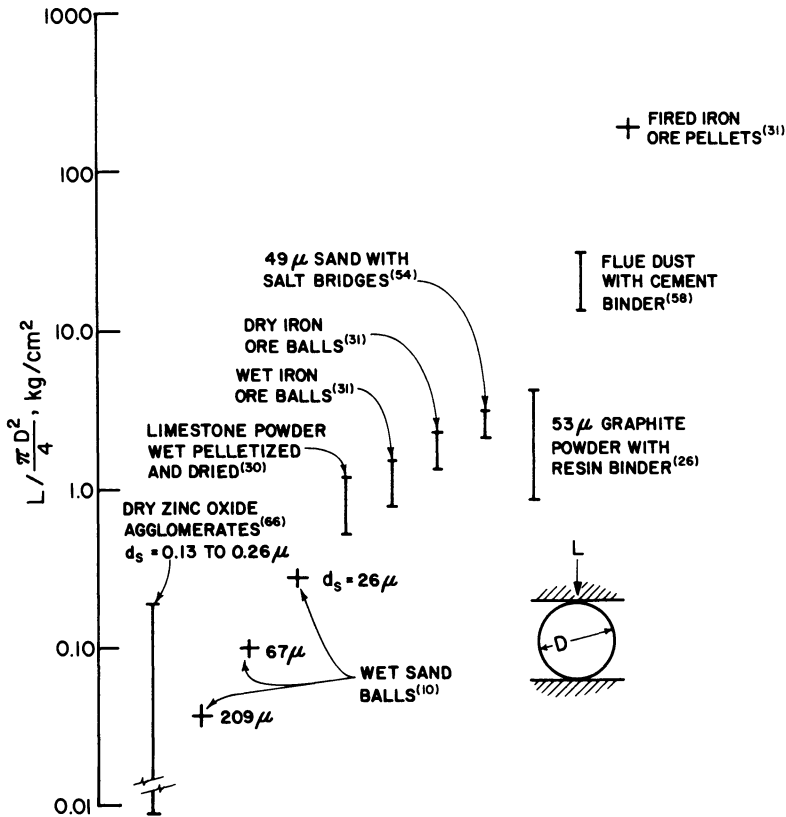


Fig. 2.9. Typical compressive strengths of various spherical agglomerates formed by tumbling.

the energy input and the pressure used and is less dependent on particle size than is the case, for example, with capillary bonding through mobile liquids.

As indicated by eqn. (4) (Section 2.1), the main variables affecting the strength of an agglomerate, other than the bonding mechanism, are the void ratio and the size of the particles from which it is composed. A number of empirical relationships have been used to correlate the effects of these variables. For example, eqn. (18) has been used [39] to represent the crushing strength of compressed tablets of constant size.

$$L = Kd^{-a} e^{-be} \quad (18)$$

where L is the crushing load, d is the particle size of powder used to prepare the tablets, e is the void ratio of the agglomerate, and K , a and b are parameters.

Equation (19) has been used [40] to correlate the compression strength of

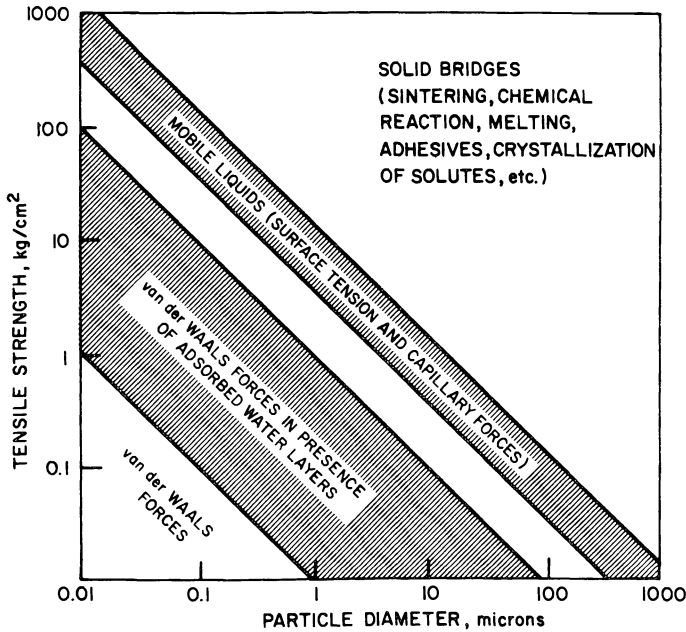


Fig. 2.10. Theoretical tensile strength of agglomerates with various bonding mechanisms [1].

chemically-bonded iron ore agglomerates.

$$\sigma_c = K \left(\frac{1-\epsilon}{\epsilon} \right)^a d^{-b} \quad (19)$$

where σ_c is the compression strength, d is the particle size, ϵ is the void ratio, and K , a and b are parameters.

4.3. Binders and lubricants

Many types [41] of additives may be used in size enlargement, including binders, lubricants, plasticizers, wetting agents, fungicides, bactericides, etc. The first two, binders and lubricants, contribute significantly to agglomerate strength, binders through particle-particle bonding and lubricants through the reduction of particle-particle friction to allow lower void fraction and closer particle contact. Lubricants are most relevant to pressure methods of size enlargement where they may also act as mold release agents. Lubricants are discussed in this context in Section 1.2 of Chapter 5.

In a critical survey of binders for fuel briquettes, Walters [42,43] notes that hundreds of binders have been patented, investigated or used. Three

TABLE 2.3

Some common binders [44].

Matrix type	Film type	Chemical type
Coal-tar pitch	Water	$\text{Ca(OH)}_2 + \text{CO}_2$
Petroleum asphalt	Sodium silicate	$\text{Ca(OH)}_2 + \text{molasses}$
Portland cement	Plastic resins	$\text{MgO} + \text{Fe}_3\text{O}_4$
Carnauba wax	Glues	$\text{MgO} + \text{MgCl}_2$
Paraffin	Starch	Sodium silicate + CaCl_2
Clay	Gums	Sodium silicate + CO_2
Dry starch	Bentonite	
Dry sugars	Tapioca	
Wood tars	Glucose	
Gilsonite	Sucrose	
	Dextrin	
	Lignosulfonates	
	Molasses	
	Alginates	

systems have been used to classify the vast number of possible binders:

1. physical state (liquid, semi-solid and solid),
2. chemical type (organic, inorganic, compound),
3. function (matrix, film and chemical types).

Regarding the latter classification [44], *matrix binders* are those in which particles are imbedded in an essentially continuous network of binding material. The amount required thus depends heavily on the void fraction of the agglomerate. *Film-type binders* are commonly used as solutions or dispersions which deposit as a film on drying. The amount required is a function of the specific surface of the particle system. *Chemical binders* develop their strength through a chemical reaction between the components of the binder or between the binder and the agglomerated material. Some common examples of each of these three types of binders are given in Table 2.3.

The selection of a binder for a given application remains a matter of experience. In Table 2.4, a rating is given for the effectiveness of a selection of binders and lubricants used in the tableting of different materials. Other reviews of binder systems used in fuel briquetting [43], ceramic [45], pharmaceutical [39,46] and foundry [47] applications are available.

Preliminary selection of binder should be made on the basis [44] of such factors as:

- (1) compatibility with the particle system, that is, ability to wet the particle surfaces,
- (2) intrinsic strength of binder and level of product strength required,
- (3) waterproof requirements in product,
- (4) contamination of the product,
- (5) binder availability and cost.

TABLE 2.4

Binders and lubricants for tableting. (Courtesy Pennwalt Corporation.)

Additives	% dry weight	Application					
		Catalyst	Ceramic	Chemical	Food	Metal powders	Pharmaceutical
<i>Binders</i>							
Agar, algin	0.5—3	—	A-B	B-C	B-C	—	B
Dextrine	1—4	—	A	B	B	—	A
Dextrose	5—20	—	—	A-B	A-B	—	A
Gelatine	1—3	—	—	A-B	A-B	—	A
Glucose	1—5	—	A	A	B	—	A
Glues	1—5	—	D	A	—	—	—
Gums	1—5	—	A	A	A	—	A
Lactose	5—20	—	—	B	B	—	A
Pitch, asphaltum	2—50	D	—	A	—	—	—
Resins	0.5—5	A	A	A	—	A	—
Salts	5—20	—	—	B	—	—	A
Silicate of soda	1—4	—	C	B	—	—	—
Starch (paste)	1—3	—	A	B	A	—	A
Sugar (sucrose)	2—20	—	—	A-B	A-B	—	A
Sulfite liquor	1—5	—	A	A	—	—	—
Waxes	2—5	—	C	C	D	A	D
Water	0.5—25	A	C	—	C	—	C
<i>Lubricants</i>							
Benzoate of soda	1—4	—	—	D	—	—	D
Boric acid	2—5	—	—	D	—	—	D
Graphite	0.25—2	A	—	A	—	B-C	—
Oils	0.25—1	—	A	C	C	C	C
Soaps	0.25—2	—	—	C	—	—	D
Starch	1—5	—	—	C	C	—	B-C
<i>Stearates:</i>							
Aluminum	0.25—2	B	B	B	—	B	—
Magnesium, calcium	0.25—2	A	A	A	A	B	A
Sodium	0.25—2	—	—	B	—	—	B
Lithium, zinc	0.25—2	A	A	A	—	A	—
Stearic acid	0.25—2	B	B	A	A	B	A
Sterotex	0.25—2	A	A	A	A	A	A
Talc	1—5	C	B	B	—	—	A
Waxes	1—5	—	B-C	C	—	B-C	D
Water	0.1—5	A	A	C	C	—	C

Effectiveness: A = excellent; B = good; C = fair; D = poor.

After rejecting obviously unsuitable binders, an experimental program using the chosen size enlargement method is generally necessary for final evaluation.

4.4. Uniformity of bonding

In theoretical treatments of agglomerate strength such as those given in Section 2, it is normally assumed that interparticle bonds are uniformly distributed throughout the agglomerate interior. This assumption is reasonable, for example in the case of wet agglomerates in the capillary or upper funicular state. Then, the binding force of capillary suction is homogeneously distributed throughout the particle assembly by means of a continuous network of pore liquid. In other situations, however, especially where solid bridging occurs, nonuniform binder distribution is expected. Examples include:

(i) Bonding by *chemical reaction* in which reactants must diffuse in from the outside of the pellet. The thickness of the bonded outer layer would then depend on, inter alia, the reaction time, the void fraction of the agglomerate and the ability of the reactants to penetrate this space.

(ii) Bonding through *pressure compaction* in which frictional effects at the die wall and between the particles lead to different levels of pressure and

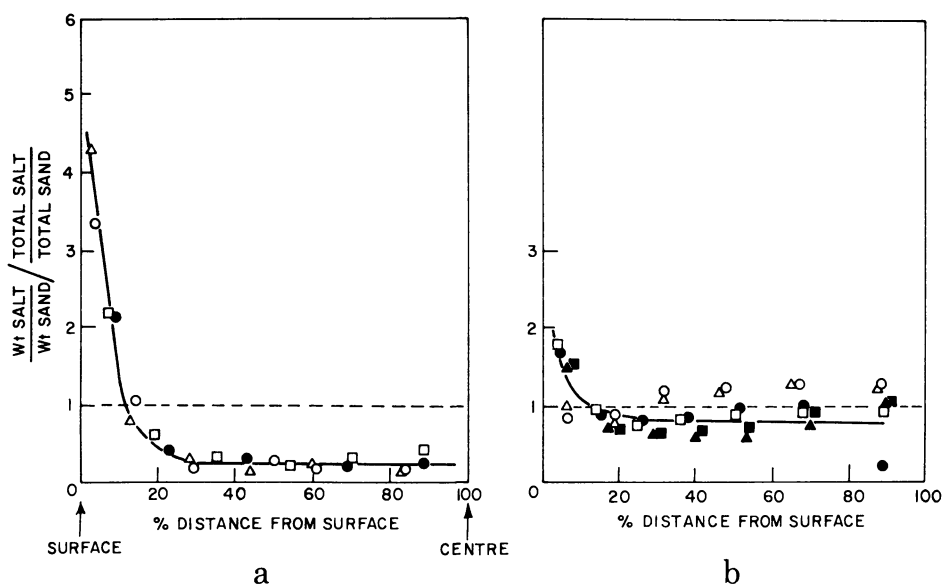


Fig. 2.11. Salt distributions in sand agglomerates [26] (31–44 μm sand; saturated aqueous solution of NaCl as bridging liquid; dried at 110°C. (a) agglomerates contained no gelling agent; (b) bridging liquid contained 10% w/v corn starch).

consequently different degrees of compaction and bonding at various locations in the agglomerate [48].

(iii) Bonding by the *deposition of dissolved materials* during the drying of green agglomerates. Throughout the capillary and funicular states of moisture in the drying agglomerate, liquid is sucked to the surface of the pellet by capillary action where it evaporates and deposits a large proportion of the total dissolved solids as a crust at or near the surface. A typical salt distribution in sand agglomerates obtained by sectioning the ball-shaped pellets is shown in Fig. 2.11a.

This crust formation or efflorescence results in undesirable agglomerate properties [26]. Strength is reduced to that of the bonded hoop at the surface, with the core of the pellet contributing little or nothing (see Section 4.5). Reduced drying rates result from blocked surface pores [3]. Caking tendencies are increased by the high concentration of soluble material in the surface.

The simplest procedure to reduce the formation of these crusts is to increase the drying rate as much as possible. It has been found [49] that the drying rate of salt-bonded agglomerates is affected strongly by the drying temperature but is little affected by the velocity of the drying air. At high drying temperatures, the liquid flow through to the surface can no longer supply liquid at the required evaporation rate. Consequently, the liquid is removed by vapour transport from progressively further inside the agglomerate as the temperature increases and more binder is deposited in situ in the core. Drying temperature cannot be raised without limit, however, since undesirable effects such as agglomerate bursting during drying and cracking on cooling occur at very high temperatures. (The effect of drying conditions on the strength of salt-bonded agglomerates is discussed further in Section 4.5.)

Other more complicated methods to reduce efflorescence during drying are available. Agglomerates can be formed in consecutive layers [50] with drying after each thin layer to yield more uniform bonding. Alternatively, drying can be arrested when a continuous network of liquid still exists in the pores as the drying state is about to pass from the funicular to the pendular. This procedure [51] allows the crust to redissolve and slowly to diffuse back into the interior of the agglomerate. A third method, the results of which are shown in Fig. 2.11b, consists of incorporating an inverse thermal gelling agent into the bridging solution. As the temperature is raised during the early stages of drying, the pore liquid is transformed into a gel, is prevented from moving to the surface during drying and the binder is consequently deposited in situ. Suitable inverse thermal gelling agents include [52]:

for aqueous bridging liquids:

- corn starch
- potato starch
- wheat flour
- methyl cellulose

for organic bridging liquids:

aluminum stearate

aluminum palmitate

aluminum linoleate

(iv) Bonding by *sintering or melting* where heat must transfer from the pellet exterior and the bond distribution depends on the heat transfer rate, the kinetics of the sintering system, the treatment time, etc. Temperature gradients during induration are accompanied by differential bond formation and shrinkage due to sintering and have been shown [53] to cause the shell and core and layered inhomogeneity defects well-known in iron ore pellets. The mechanism of the process is illustrated in Fig. 2.12 which represents a pellet under conditions of high heat transfer rate to the surface. Under these conditions the outer layers of the pellet are considered to have reached sintering temperature and considerable shrinkage has occurred while the interior is at lower temperatures where bonding and shrinkage are negligible. The pellet core is effectively incompressible so that the surface shrinkage is accompanied by force components A in the diagram which result in the formation of surface cracks. Wynnyckyj and McCurdy [53] have calculated the degree of shrinkage expected within a pellet as a function of radial position during the sintering of a commercial magnetite concentrate in fully-oxidized form. Fig. 2.13 shows some of their results under “severe” heat transfer conditions where it is seen that the shrinkage at the pellet surface is much larger than that at the centre and the effect is increased as pellet size increases. The correspondence of this effect with the separation into concentric layers suggested in Fig. 2.12 is evident. These authors also derived a “propensity index” from their theoretical calculations to gage the probability of forming layered inhomogeneity defects under various conditions. Industrial experi-

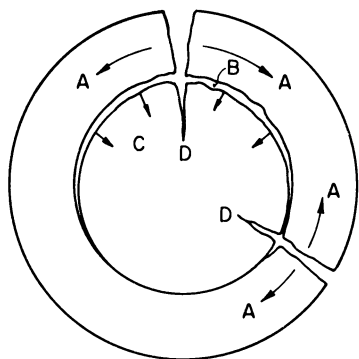


Fig. 2.12. Model of the formation of concentric shells during sintering under non-steady state temperature conditions [53]. (Copyright American Society for Metals and The Metallurgical Society of AIME, 1974.)

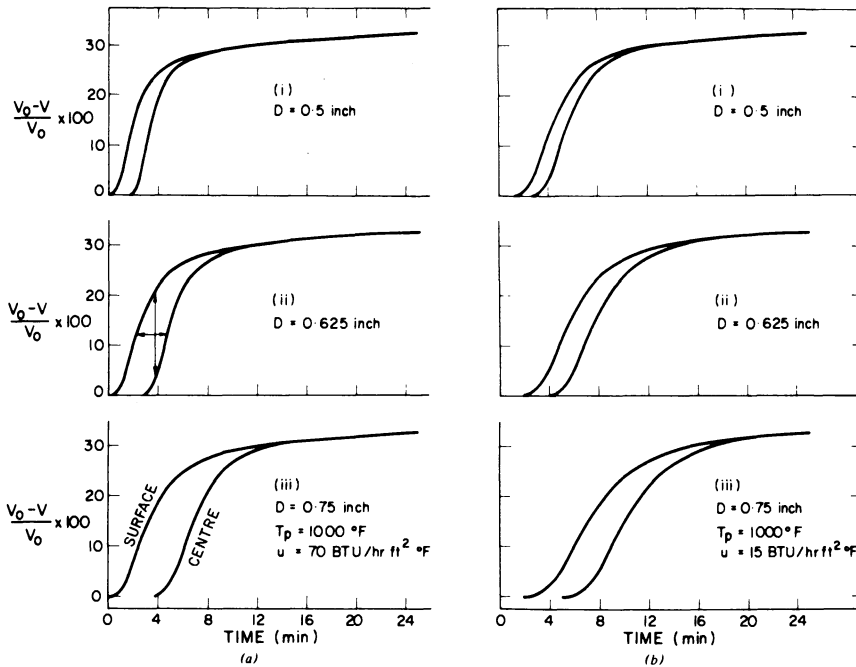


Fig. 2.13. Comparison of shrinkage parameter, $(V_0 - V)/V_0$, as a function of time at the surface and centre of pellets of different sizes for two different heat transfer conditions [53]. (a) $U = 70 \text{ Btu}/(\text{h ft}^2 \text{ } ^\circ\text{F})$; (b) $15 \text{ Btu}/(\text{h ft}^2 \text{ } ^\circ\text{F})$. (Copyright American Society for Metals and The Metallurgical Society of AIME, 1974.)

ence that more homogeneous pellet bonding may be obtained by reducing pellet size and that gradual heating to the firing temperature is important was clearly corroborated by this work.

4.5. Bond distribution and the size-strength relationship

Common practice to normalize the compressive strengths of spherical agglomerates of different sizes is to divide individual loads at failure by $(\text{pellet diameter})^2$ (cf. Section 4.1). This treatment of pellet strength data, however, assumes a uniform radial distribution of bonds within the agglomerates. That is, in the generalized relationship between load at failure and agglomerate diameter,

$$L = KD^n \quad (20)$$

when n has a value of 2, the binding forces are considered to be exerted uniformly over the whole cross-section. On the other hand, when most of the binding forces are concentrated as a surface crust, the binding cross-section

TABLE 2.5

Crushing strength of graphite agglomerates [26] (minus 300 mesh graphite, balled with organic bridging liquid and dried at 75°C).

Bridging liquid	Dissolved binder	Gelling agent	Correlating equation
Toluene	20% w/v petroleum resin	none	$L = 12.9 D^{1.1}$
Toluene	none	20% w/v aluminum stearate	$L = 10 D^{2.2}$
Toluene	20% w/v petroleum resin	10% w/v aluminum stearate	$L = 49 D^{1.75}$

would be directly related to the pellet circumference, and n has a value close to 1. A simple geometric model has been proposed [54] to show how the exponent n might assume values ranging between 1 and 2 for bonding situations intermediate between the two extremes of "surface-hardened" and uniformly-bonded agglomerates.

The relationship between the value of the exponent n in the compressive strength correlation and the uniformity of bonding is demonstrated by the data in Table 2.5. When petroleum resin dissolved in toluene is used as a bridging liquid to form agglomerates from graphite powder, the dried agglomerates form a surface crust of resin binder and their compressive strengths are correlated by eqn. (20) with n close to unity. When an inverse thermal gelling agent is present in the bridging liquid (cf. Section 4.4), the binder is held in situ throughout the interior during drying, and the index n approaches a value of 2.

TABLE 2.6

Crushing strength of salt-bonded agglomerates formed at various drying temperatures [54] (49 μm sand balled with aqueous sodium chloride solutions).

Drying temperature (°C)	Sodium chloride concentration (g/cm ³)		
	0.35	0.23	0.11
<i>n</i> values (eqn. 20)			
60	1.68	1.53	1.48
110	1.42	1.54	1.47
220	2.01	2.03	2.20
300	1.97	2.08	2.16
<i>K</i> values, lb/(in.) ⁿ (eqn. 20)			
60	19.8	16.5	14.6
110	11.3	18.8	15.7
220	35.6	41.3	27.5
300	37.4	43.0	24.4

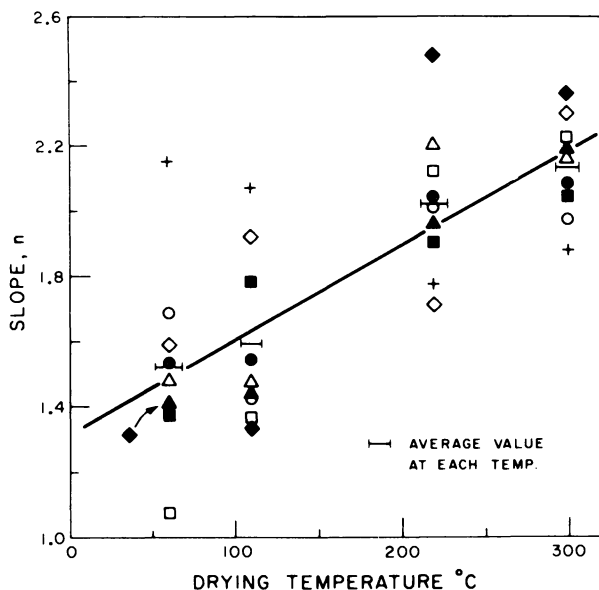


Fig. 2.14. Exponent n from eqn. (20) applied to crushing strength of salt-bonded agglomerates, as a function of drying temperature [54] (agglomerates of sands of $49\ \mu\text{m}$, $103\ \mu\text{m}$ and $208\ \mu\text{m}$ diameter ballled with aqueous sodium chloride solutions of various concentrations).

The compressive strength—agglomerate diameter relationship correlated by means of eqn. (20) has also been used [54] to interpret the mode of crystallization and distribution of salt bridges in dried sand agglomerates which had been wet ballled using a salt solution as bridging liquid. Agglomerates ranging in size from $\frac{1}{8}$ in. (3.2 mm) to $\frac{7}{8}$ in. (22.2 mm) diameter formed from $49\ \mu\text{m}$, $103\ \mu\text{m}$ and $208\ \mu\text{m}$ sands using aqueous bridging liquids with three different salt concentrations (0.35 , 0.24 and $0.11\ \text{g NaCl/cm}^3$) were dried at various temperature levels. The least squares values of the parameters n and K from eqn. (20) are plotted in Fig. 2.14 and listed in Table 2.6, respectively. Fig. 2.14 indicates that there is an increase in the exponent n , that is, a more uniform distribution of crystal bonds according to the above discussion, as the drying temperature is raised. These results are consistent with previous studies [3,51,69] in which binder distribution was determined by sectioning and chemical analysis. It was found that the proportion of salt deposited in the core increases and crust formation becomes less pronounced as the temperature is raised. The agglomerate strength factors, K , in Table 2.6 generally increase with drying temperature and with the salt concentration. There is a good indication, however, that neither of these two variables can be increased without limit to improve agglomerate strength. An excessive drying tempera-

ture or a too high salt concentration may cause a peak in strength or even a reduction. A high drying temperature and thus drying rate lead to deposition of finer crystals with greater bonding strength but excessively high drying temperatures may cause weaknesses due to cracks which form on cooling. In the case of salt concentration, increasing salt content produces more interparticle bonds and greater strength, but excessive salt concentration can lead to efflorescence of the bonding material with reduced drying rates and thus weaker interparticle crystal bonds. Excessive efflorescence may also result in "bubble" defects in the outer crust and reduced compressive strength due to these flaws.

Equation (20) and the deviation of the index n from the "uniform-bonding" value of 2 have been used to interpret compressive strength data for wet and dry iron ore balls [55] and for fertilizer granules [56].

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AGITATION METHODS — TUMBLING AGGLOMERATION

Fine powders can readily be formed into granular masses by admixture with a liquid phase followed by suitable agitation. The liquid and solid phases are thus brought into intimate contact to develop binding forces and cause agglomeration. The most common wetting phase is water or an aqueous solution with capillary binding forces being developed in the agglomerates. Other liquids and binding mechanisms may be utilized, however, with attendant development of viscous liquid, solid, van der Waals and other binding mechanisms. The most frequent agitation means is the tumbling, rolling, cascading action of disc, cone or drum devices. This type of equipment is considered in this chapter, while other types of mixers are examined in Chapter 4. Some fundamental principles applicable to all agitation methods are first discussed.

1. Fundamental aspects

1.1. Agglomerate nucleation and growth [1–6]

In wet agglomeration, the initial moist feed material is in the form of loose fluffy aggregates which form into nuclei during the early stages of agitation. The surface tension of the binding liquid is responsible for this phenomenon and the reduction in the total surface free energy of the system accompanying the decreased air-liquid interfacial area is the driving force (see Fig. 3.1). This nucleation phenomenon is most evident with very fine particles (e.g. minus 325 mesh) and may account for agglomerate growth up to 1 or 2 mm diameter, depending on the conditions.

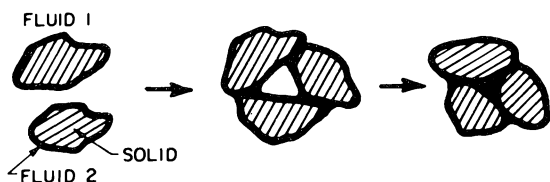


Fig. 3.1. Schematic representation of the formation of nuclei through reduction in the total air-water interface [6].

Beyond the nucleation stage, size change in the system can occur by a number of mechanisms as depicted in Fig. 3.2. The prevailing mechanism depends on such factors as the feed particle size and other solid properties, liquid surface tension and viscosity and the mode of operation (batch or continuous). After nucleation has occurred, the predominating growth mechanisms are:

(1) coalescence, and

(2) layering of either feed particles or fines from the degradation (shattering, crushing or breaking, attrition) of established agglomerates.

Both coalescence and layering can occur simultaneously in a system but, in general, one mechanism will predominate.

In *batch systems* involving relatively weak agglomerates larger than 2 to 6 mm diameter, growth occurs by degradation of the smaller agglomerates with the fines then layering onto the remaining agglomerates. With water as the liquid phase, closely-sized powders of average size greater than about $70\ \mu\text{m}$ typically grow in this way in balling drums. By contrast, the coalescence mechanism is favoured with finer powders (e.g. minus 325 mesh) of wider size distribution and larger surface area. Agglomerates of these powders not only resist degradation due to their larger strength but also undergo a consolidation period after nucleation in which excess liquid phase is

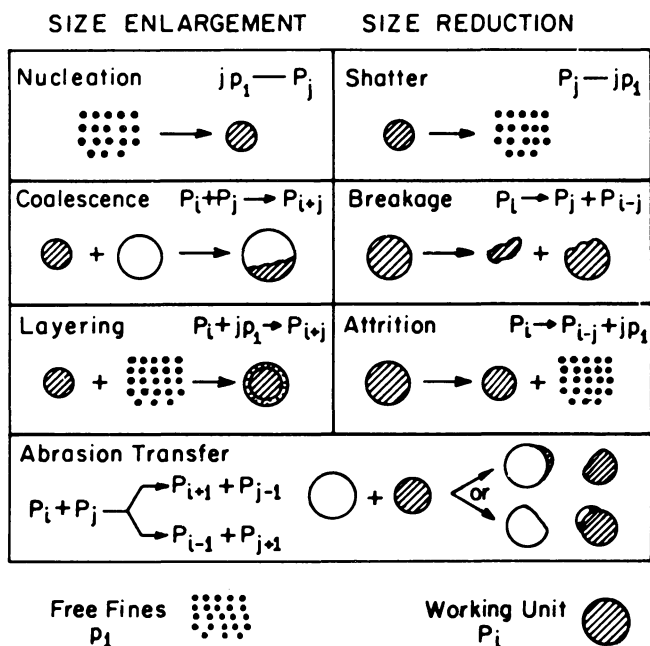


Fig. 3.2. Formal representation of mechanisms of size change in particulate processes [6].

exuded at the surface. This excess moisture is available to form bridges between agglomerates and can also lead to plasticity in the surfaces, both of which encourage coalescence. The rate of growth by coalescence for larger agglomerates (e.g. larger than 5 or 6 mm diameter) is inherently slow, however, since the separating torque on a coalescing twin reduces the efficiency of coalescence as size increases.

In *continuous systems*, recycled undersize material or agglomerates formed by coalescence of the fresh feed act as nuclei. However, since the fine feed particles can form stronger bonds with the nuclei particles than with each other, the majority of the feed material would be expected to simply layer onto the established agglomerates rather than to coalesce with other fine particles.

Agglomerates formed by coalescence and by layering can usually be distinguished by their appearance. Layered granules tend to a smooth spherical shape and examination of their interior cross sections reveals a layered "onion skin" construction. Coalesced granules tend to a more irregular spheroidal shape and often exhibit a rough "raspberry"-type surface.

1.2. Influence of agglomerate strength: maximum feed particle size

It has already been noted that agglomerate strength is important in determining the mechanism by which size enlargement by agitation occurs. Relatively weak agglomerates can grow in size by the destruction of the smallest and weakest in a batch with the fragments layering onto the surviving agglomerates.

Agglomerate strength plays an even more fundamental role in that it determines whether or not nucleation and growth can occur at all in an agitated moist powder. To survive and grow, the cohesive forces present in a nucleus which are responsible for its formation must be able to withstand the destructive agitation forces of its environment. Once formed, the final size reached by the agglomerate represents a balance between these same cohesive and destructive forces.

In quantitative terms, the cohesive strength of an agglomerate is reasonably well-defined by eqn. (13) and eqn. (14) of Chapter 2. The destructive agitation forces, however, are generally not quantitatively understood although empirical relationships to represent this balance between destructive and cohesive forces for particular pieces of equipment and particular powder systems are available. Thus, in a study [2] of the balling of uniformly-sized sands using water and aqueous alcohol solutions to wet the powders tumbled in a small laboratory drum operated at 37% of its critical speed, satisfactory balling was obtained only if the ratio of surface tension of liquid to particle diameter exceeded a minimum value (cf. eqn. (13) and eqn. (14) of Chapter 2):

$$\frac{\sigma}{d} \geq 4.6 \times 10^3 \frac{\text{dynes}}{\text{cm}^2} \quad (1)$$

The addition of fines to a powder system allows a larger top size of particle to be agglomerated due to the attendant increase in cohesive forces caused by a decrease in surface mean particle diameter and increase in agglomerate density. Thus, although eqn. (1) indicates a top size of about $150\text{ }\mu\text{m}$ for monosized particles with aqueous binders, the top size of feed for industrial disc pelletizers is usually higher at 30 to 50 mesh (300 to $600\text{ }\mu\text{m}$) with the provision that at least 25% should be finer than 200 mesh ($75\text{ }\mu\text{m}$) [7]. Other liquids with surface tensions lower than that of water, or liquid/solid systems in which the particle surface is imperfectly wetted, require finer particle sizes to make successful balling possible.

The above particle size specifications apply to the formation of relatively large agglomerates (say, larger than 3 or 4 mm) formed under the relatively gentle tumbling action of a balling drum or disc. More intense agitation, such as that produced in a high speed paddle mixer, and coarser feed particle sizes can produce agglomerates but they are of a smaller size. An example is fertilizer granules produced in pugmills using feed particles whose average diameter may be in excess of 1 mm [8].

1.3. Bridging liquid requirements

Agglomerates formed by wet pelletization are generally considered to be in the capillary state, that is the internal pore structure is just saturated with binding liquid. Eqn. (2) can then be derived for the liquid content of such an agglomerate on a wet basis:

$$W = \frac{\epsilon\rho_L}{\epsilon\rho_L + (1 - \epsilon)\rho_S} \quad (2)$$

where W is the weight fraction of liquid (wet basis), ϵ is its void fraction, and ρ_L and ρ_S are particle and liquid densities, respectively.

Equation (2) has been fitted [9] to a wide variety of literature data in which solid density ranged from about 1 to 6 g/cm^3 while liquid densities were generally close to 1 g/cm^3 . The following relationships were found for average feed particle diameters $<30\text{ }\mu\text{m}$:

$$W = \frac{1}{1 + 1.85(\rho_S/\rho_L)} \quad (3)$$

for averaged feed particle diameters $>30\text{ }\mu\text{m}$:

$$W = \frac{1}{1 + 2.17(\rho_S/\rho_L)} \quad (4)$$

Porosity information was generally not available for the data used, hence this effect is included in the fitted constants in eqn. (3) and (4). The tendency to higher pore volumes in agglomerates made from fine particles accounts for the higher liquid contents predicted by eqn. (3).

Relationships (3) and (4) predict the liquid content for agglomeration to within an accuracy of only about 30%. A generalized equation of greater accuracy is not available due to the large number of factors which can influence the value of W . These include:

- (a) the wetting properties of the liquid/solid system,
- (b) the solubility of the solid components and the extent to which solubility equilibrium is reached in the system,
- (c) agitation conditions and mixing time,
- (d) liquid properties (viscosity, surface tension, etc.),
- (e) solid properties (shape, size, roughness of particles; ability to adsorb water or form combined water),
- (f) the fraction of the internal pores actually occupied by liquid (up to 30 or 40% may contain trapped gas).

Some attempts have been made to include more of these effects in equations to predict optimum moisture content for balling. For example, the following equation has been derived [10] for the optimum moisture content of green iron ore pellets:

$$W = \frac{100AK}{1 + \frac{V_s d_s}{V_p d_w + (d_{\text{comb}} - d_w) S_m l_0 V_s}} \quad (5)$$

where W is weight % moisture in a pellet, V_p is the pore volume in a pellet (cm^3), V_s is the volume of solid phase in the pellet (cm^3), d_{comb} is the density of combined water at the pellet temperature (g/cm^3), d_w is the density of the free water (g/cm^3), S_m is the specific surface of the mix (cm^2/cm^3), l_0 is the combined water film thickness (cm), and d_s is the density of the solid phase (g/cm^3).

Variables d_{comb} and l_0 have values of about $1.2 \text{ g}/\text{cm}^3$ and $21 \times 10^{-6} \text{ cm}$ for iron ores. Parameter A depends on the extent to which the particles are hydrophilic and has a value of 1.0 to 1.06 for silica contents of 3–4% and 12–14% in 65% total Fe magnetite ores, respectively. Parameter K depends on mixing conditions in the pelletizer and ranges from 0.65 to 0.7.

It should be noted that it is the total *free liquid* phase present during granulation which controls the agglomeration process. If the feed powder contains material which can combine with some of the added liquid (e.g. bentonite [6]), this must be taken into account in calculating the operative amount of liquid during the process. Similarly, soluble constituents can increase the total amount of liquid above that originally added to the feed powder. This phenomenon introduces a marked temperature dependence into the required moisture content as demonstrated by the agglomeration curves for spray-dried coffee and fertilizer mix in Fig. 3.3 and 3.4, respectively. Correlations such as these refer only to one particular feed chemical composition produced from a particular particle size distribution in a particular type of mixing system.

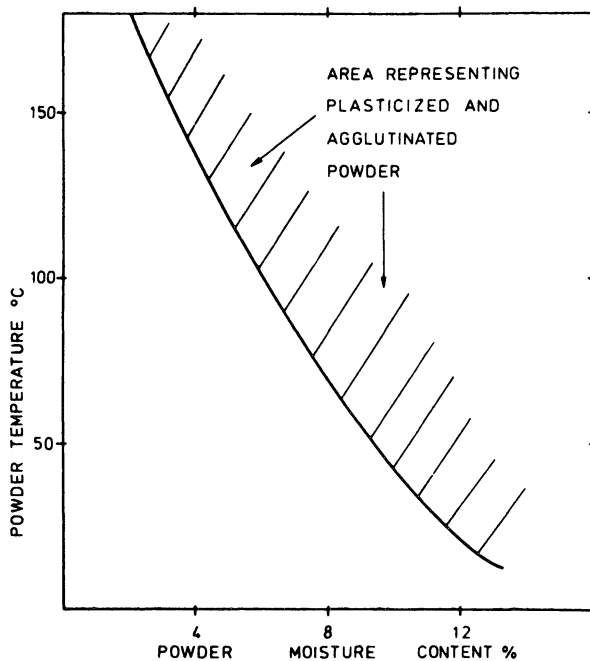


Fig. 3.3. Typical agglutination curve for instant spray-dried coffee [34].

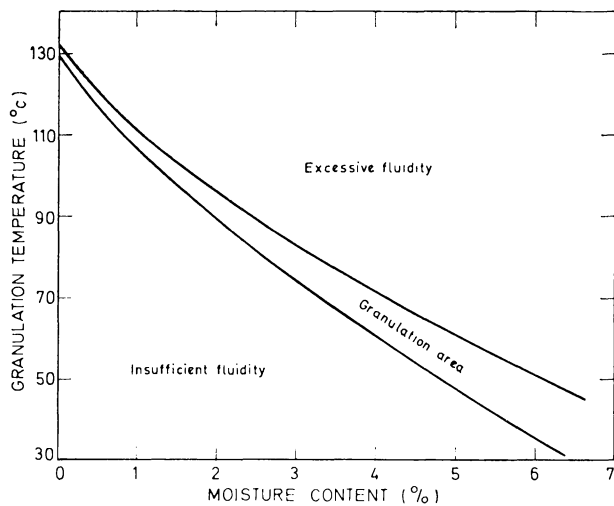


Fig. 3.4. Typical temperature-moisture relationship for granulation of one particular fertilizer composition [35] (composition and particle size not specified).

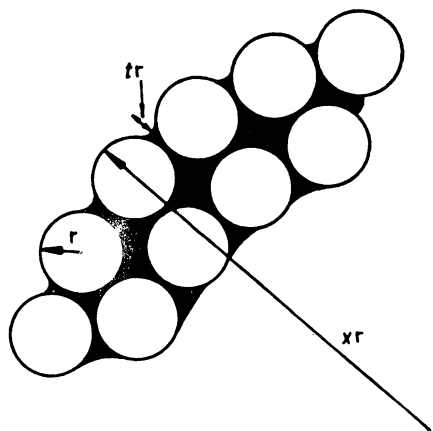


Fig. 3.5. Representation of "dry" granule surface in granulation model of Sherrington [8].

The bridging liquid necessary to form small granules (e.g. 1 or 2 mm diameter) has been found [8] to be much less than that for larger balls and much less than that theoretically necessary to saturate the feed powder in a compacted state. The reason for this can be appreciated with the aid of Fig. 3.5. While the interior of both small and large agglomerates is saturated with liquid, the surface is relatively dry due to the withdrawal of liquid into the surface pores to develop negative capillary pressure. Since small granules have a larger surface/volume ratio than large ones, they also contain a smaller volume of liquid relative to their solids content than do larger ones. Geometrical considerations lead to the following equation for the liquid requirements of a "surface-dry" agglomerate:

$$y = k \left(1 - \frac{3td}{D} \right) \quad (6)$$

where y is the ratio of liquid phase to solid phase, k is the ratio of void volume to solid volume, t is a surface withdrawal parameter, and D and d are the agglomerate and feed particle diameters, respectively.

In eqn. (6), it will be noted that as D/d becomes large, y approaches k , indicating (consistent with the above discussion) that for large agglomerates the amount of liquid equals that necessary to saturate the voids. The greatest value of eqn. (6) is not in the absolute values of y which it predicts, but in providing the functional relationship between liquid requirements and agglomerate size for smaller agglomerates. The original work [8] showed that k had values much less than those calculated from packed bulk density measurements, indicating that a large portion of the pore space in the agglomerates may be filled with air. By definition, t should be a reasonable fractional number and was in fact found to be in the range 0.22 to 0.57. Values

TABLE 3.1

Moisture requirements for balling a variety of materials

Raw material	Approximate size analyses of raw material, less than indicated mesh	Moisture content of balled product (% H ₂ O)
Precipitated calcium carbonate	200	29.5—32.1
Hydrated lime	325	25.7—26.6
Pulverized coal	48	20.8—22.1
Calcined ammonium metavanadate	200	20.9—21.8
Lead—zinc concentrate	20	6.9— 7.2
Iron pyrite calcine	100	12.2—12.8
Specular hematite concentrate	150	9.4— 9.9
Taconite concentrate	150	9.2—10.1
Magnetic concentrate	325	9.8—10.2
Direct shipping open pit ores	10	10.3—10.9
Underground iron ore	0.25 in.	10.4—10.7
Basic oxygen converter fume	1 μ m	9.2— 9.6
Raw cement meal	150	13.0—13.9
Utilities—fly ash	150	24.9—25.8
Fly ash—sewage sludge composite	150	25.7—27.1
Fly ash—clay slurry composite	150	22.4—24.9
Coal—limestone composite	100	21.3—22.8
Coal—iron ore composite	48	12.8—13.9
Iron ore—limestone composite	100	9.7—10.9
Coal—iron ore—limestone composite	14	13.3—14.8

Courtesy McDowell Wellman Company.

of k and t for a particular agglomeration system, however, must be determined experimentally.

Useful indications of binding liquid requirements can be obtained from eqn. (3) to (6) or from quoted data, such as that in Table 3.1. In general, most powders require 10 to 20% moisture for balling, with a maximum of about 30%. For very accurate data on the optimum liquid content to agglomerate a particular powder, experiments with the material are necessary.

1.4. Kinetics of growth

It is obviously desirable for high throughput to increase the rate of growth of agglomerates in a particular apparatus to the maximum compatible with acceptable product properties. Excessive growth rates may lead to non-uniform product shape, high internal porosities and poor agglomerate strength while controlled growth encourages better-formed products.

Batch experimentation in laboratory tumbling agglomerators, often with

model systems, has shown [11–13] that prime factors influencing the rate of agglomerate growth are:

- (1) bridging liquid content of the feed powder,
- (2) speed of rotation of drum,
- (3) drum loading,
- (4) size and size distribution of feed,
- (5) liquid surface tension,
- (6) presence of seeds or nuclei.

The mean granule size for larger agglomerates growing by crushing and layering generally increases linearly [11] with the duration of tumbling. The slope of such plots readily yields a measure of the rate of growth. With smaller agglomerates growing by coalescence, non-linear size-time relationships are found which can be treated to yield a coalescence rate function [14].

The rate of agglomerate growth depends upon their ability to deform/degrade and to coalesce/layer upon impact with one another under the agitation conditions. In wet agglomeration, growth occurs due to the “plasticity” and “surface wetness” of the granules which contain, as noted above, somewhat more or less than the theoretical saturation liquid level. Small variations in liquid content cause large changes in agglomerate plasticity and the ability to deform and coalesce through surface moisture bonds. As a result, the agglomerate growth rate is highly sensitive to the *bridging liquid content* and in all cases responds in a manner similar to that indicated in Fig. 3.6. For changes in bridging liquid content of the order of only 20 or 30%, a

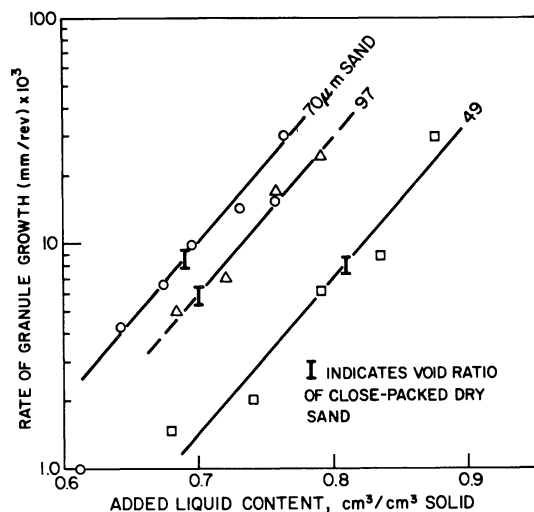


Fig. 3.6. Semi-logarithmic plot of agglomerate growth rate in laboratory balling drum as a function of moisture content. (Data of Capes [2]; sand particles of average sizes 70 μm (○), 97 μm (△) and 49 μm (□).)

many-fold change in the rate of granule growth is found. The semi-logarithmic relationship in Fig. 3.6 means that a simple equation can represent [1,14] the variation of growth rate constant, k , with the moisture content, W :

$$k = a \exp(bW) \quad (7)$$

As seen in Fig. 3.6 and consistent with the discussion in the previous section, the theoretical bridging liquid requirements to saturate the powder in a close-packed state lies at intermediate rates of growth. In view of the extreme sensitivity of the growth to liquid content, however, and the previously-indicated inability to predict accurately optimum liquid levels for agglomeration, the constants a and b in eqn. (7) for a particular powder-liquid system and specific agitation conditions must be determined by experiment.

The rate of growth in terms of size change per unit drum revolution, has been found [11,12] to be independent of the *speed of rotation* below about 35% of the critical speed of the drum. Of course, longer times are required to grow agglomerates of a given size as the speed of rotation is reduced. Up to a *drum loading* of about 6% by volume, the growth rate increased in proportion to the loading for coarser feeds producing relatively weak agglomerates, while the drum loading had no effect on the growth rate of agglomerates formed from fine taconite particles. It was suggested that these latter agglomerates, being stronger, are therefore less affected by changes in the size of load pressing down during tumbling. Excessive speeds and loadings can produce very rapid, uncontrolled growth due to cascading and the pulverizing action of the larger agglomerates. These phenomena are undesirable since agglomerate quality suffers under these conditions.

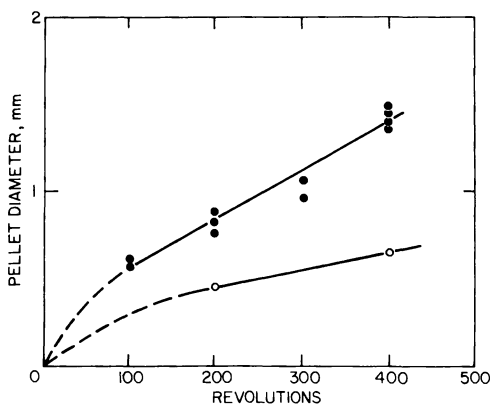


Fig. 3.7. Effect of particle size and size distribution on the growth rate of iron ore pellets in a laboratory balling drum [13]. (By permission The Institution of Mining and Metallurgy.) [Hematite ore, minus 300 mesh (○) and minus 120 plus 200 mesh (●).]

Since agglomerate growth depends on deformation and/or degradation of colliding entities, granule strength is a fundamental determinant of the growth rate. Lower strength is expected to increase agglomerate deformation and fragmentation and hence, to increase the growth rate. According to eqn. (13) of Chapter 2, agglomerate strength decreases as *particle size* increases, *particle size distribution* becomes narrower (larger porosity) and the binding *liquid surface tension* decreases and all of these changes in a wet granulation system are found, as expected, to increase the rate of agglomerate growth. The data in Fig. 3.7 illustrate the point with iron ore in which pellets are formed faster with the coarser, closely-sized feed ($-120 + 200$ mesh) compared with -300 mesh feed of finer size and wider size distribution. In the same study [13], it was also reported that the growth rate of iron ore pellets increased when water was replaced by a detergent solution of lower surface tension as bridging liquid.

The kinetics of a granulation process can be dramatically improved by the addition of recycled "seed" agglomerates. These nuclei readily pick up the fine feed material in layer growth since the pendular liquid bond between two particles increases in strength as the size difference between the particles becomes larger [15]. It has been suggested [16] that the coalescence proba-

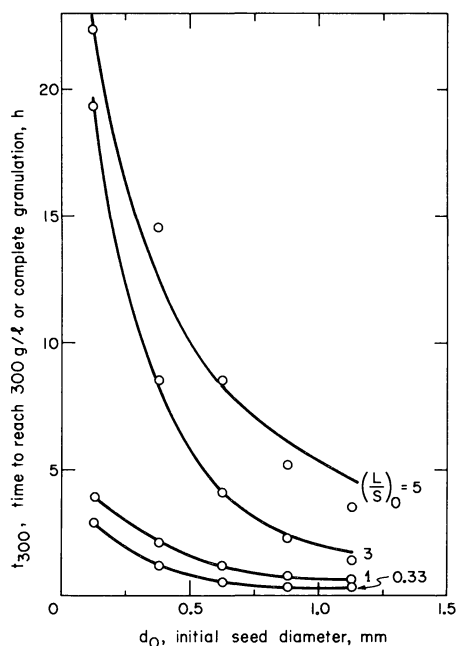


Fig. 3.8. Effect of amount of seeds added and their size on time required to reach a bulk density of 300 g/l during dry granulation of carbon black in a laboratory drum [17,36]. (Carbon black pellets used as seeds; $(L/S)_0$ is the initial volume ratio of fines to seeds.)

bility between two entities of size D and d can be represented by $a/(Dd)^b$ where a and b are fixed. It is evident that for a recycled seed of constant size D , the coalescence probability improves as the layering fines decrease in size, d .

This effect of seed addition is illustrated in Fig. 3.8 for the dry granulation of carbon black powder tumbled in a drum [17]. The time required to convert the whole load to granules is reduced as the proportion of recycled agglomerates and their size are increased. For the dry granulation of zinc oxide powders, Meissner et al. [18] found that the initial rate of fines disappearance, $(dW/dN)_{N=0}$, in the presence of seed granules is given by:

$$\left(\frac{dW}{dN}\right)_{N=0} = -K \frac{D_B^2 \rho_B V_B^3}{f(\rho_F)} \quad (8)$$

where D_B is the seed diameter, ρ_B is the seed density, V_B is the volume fraction seeds and $f(\rho_F)$ is a function of the fines density.

1.5. Agglomerate size distributions

Various studies [1,2,4] have shown that the agglomeration of moist powders by tumbling can lead to self-preserving size distributions. That is, the ratio of the spread of granule sizes to the mean size at any given time is approximately constant during *batch* balling.

Although a number of mathematical relationships can be used to fit agglomerate size distributions, an examination [19] of a number of published studies of laboratory-scale balling indicated that the log-normal distribution provides a very satisfactory representation. The ratio of the spread of agglomerate sizes (defined as the difference between the sizes at the 84% and the 16% population cuts) to the mean agglomerate size was found to be in the range 0.3 to 0.8 in most cases, the larger values corresponding to the finer feed powders.

Self-preserving size distributions are found during batch balling where growth occurs by coalescence or by degradation of smaller agglomerates with the fines then layering onto surviving agglomerates (see Section 1.1). For *continuous* systems in which layering of feed onto established agglomerates is favoured, self-preservation of the size distribution is not expected [20].

2. Equipment

The earliest agglomeration equipment consisted simply of solid-liquid mixers and, in common with newer designs, provided the mechanical means by which particles were brought into contact and densified into larger entities as the cohesive forces came into operation. Various other types of equip-

ment for proportioning, dry mixing, materials handling, grinding, drying, cooling and sizing are important adjuncts to the agglomeration process [21], but these will not be considered here.

A wide variety of agitation equipment is used industrially for agglomeration indicating that the process is still developing in methodology and applications. The most important type of apparatus, in terms of tonnages produced, is that in which a tumbling, rolling action is imparted to yield spheroidal-shaped agglomerates. The two most common devices are the rotary drum or cylinder and the inclined disc or pan, but variations of these basic machines are also used.

2.1. Inclined discs

These machines consist basically of a tilted rotating plate equipped with a rim to contain the agglomerating charge (Fig. 3.9). Solids are fed continuously from above onto the central part of the disc and discharge as agglomerates over the rim. Moisture or other binding agents can be added at various locations on the plate surface by means of the sprays provided. To promote lifting and rolling of the charge and to protect the disc surfaces, a lining of expanded metal or an abrasive coating is provided. Adjustable scrapers and plows which may oscillate mechanically maintain a uniform protective layer of product over the surface and also control the flow pattern of material on the disc. Pan angle is adjustable from 40° to 70° to the horizontal to obtain the best results and both constant-speed and variable-speed motors are available as disc drives. Dust covers are fitted when required.

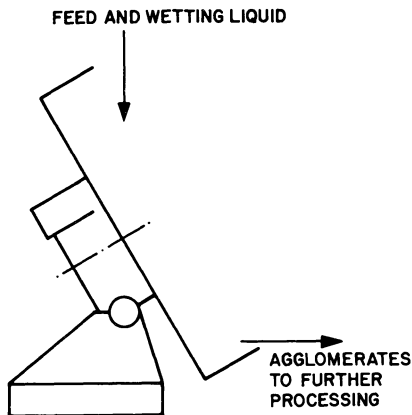


Fig. 3.9. Schematic diagram of an inclined disc agglomerator. (In many applications, subsequent screening is not required because of size classification on the disc; see Fig. 3.10.)

2.1.1. Size classification effect

A most important feature of the inclined disc agglomerator is its size separating ability (Fig. 3.10). Feed particles and the smaller agglomerates are able to sift down to the bottom of the tumbling load where, because of their high coefficient of friction, they are carried to the highest part of the disc before rolling downwards in an even stream. Larger agglomerates remain closer to the top of the bed, where they travel shorter paths. In continuous operation the largest agglomerates are discharged from the top of the bed over the rim, while smaller ones and feed fines are retained for further growth.

This classifying effect means that in many cases the product size is sufficiently uniform not to require further screening. Where strict limits are placed on product size, such as in iron ore pelletizing [22], screens may be used following the disc and commonly result in recycle rates of the order of 15%. A patented rotating comb-like sorting and ejecting unit can be fitted to Eirich [23] pelletizing pans to produce more uniform agglomerate size.

The fact that agglomerates and particles of different sizes follow different paths in the disc load means that the stage of growth at which agglomerates are wetted can be chosen, lending flexibility to the machine operation. The positioning of the sprays and the point of feed addition on the disc surface is thus of considerable importance. Direct spraying onto incoming feed particles and onto the fines in their trajectory encourages coalescence and nucleation of new agglomerates. Wetting of established agglomerates favours the layering of dry fines onto them. Spray positioning requires experimental trials to obtain the best result for a given feed material and product requirements.

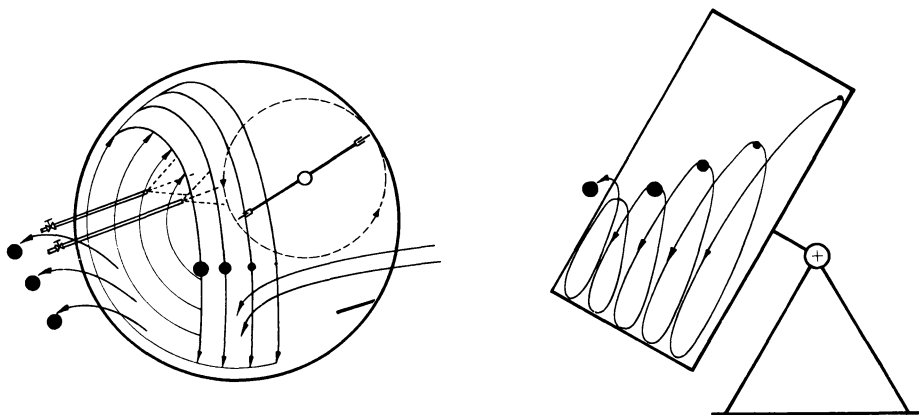


Fig. 3.10. Flow of material in a balling pan, indicating segregation and discharge of the largest agglomerates. (Courtesy Maschinenfabrik Gustav Eirich.)

2.1.2. System and operating variables

The influence of a number of the system variables relating to powder and liquid properties, etc., has already been discussed in Section 1 above. With proper control of these variables and of the pan operating conditions, it is possible within limits to influence agglomerate properties such as shape, size and porosity. A discussion of such factors has been given by Pietsch [24] and by Ball [25].

A guide to disc dimensions (diameter, depth), speed of rotation, throughput and power input is given in Section 2.1.3 below for “normal” or “average” operating conditions. For a specific powder/binder feed material, however, optimization of disc conditions requires trials with the actual machine. Only a qualitative guide to expected effects of some variables can be given here.

Agglomerate shape is influenced by the quantity of spray liquid added, the fineness of the feed particles and the mode of growth as determined by the positioning of the liquid sprays. With too much binding liquid, agglomerates become very plastic, tend to coalesce and form irregular, “raspberry”-type entities. This occurs especially with very fine particles such as minus 325 mesh material. Smoother, more spherical agglomerates are encouraged by layer growth obtained when, as noted in Section 1.1 above, established agglomerates in the pan are wetted by the sprays and dry fines are rolled onto their surfaces.

Agglomerate size is controlled primarily by retention time on the disc and the amount of added binder liquid. The relationship between these variables is shown qualitatively in Fig. 3.11. Residence time and hence pellet size can

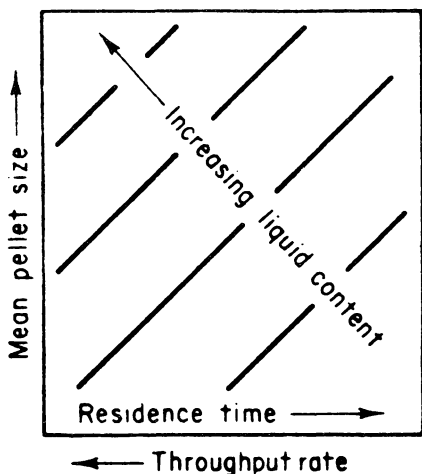


Fig. 3.11. Relationship between mean pellet size and residence time (throughput rate) as a function of liquid content for pan agglomerators [24,37].

be increased by decreasing the feed rate (throughput) and, in addition, by increasing pan depth (rim height) and decreasing the angle of inclination with the horizontal. Residence time can also be lengthened by increasing disc speed. A larger proportion of the available surface is used as the material is carried to higher trajectories and thus solids hold-up increases. Excessive speed can, however, result in agglomerate degradation and breakage if the intensity of agitation becomes too large.

Agglomerate porosity is influenced by the magnitude of the mechanical forces in the pan, by the retention time and by the amount of liquid added. If too little liquid is added, the agglomerates are too brittle and lack sufficient plasticity to compact properly during the tumbling. With optimal liquid content, increasing pan diameter increases the height of fall of the agglomerates and produces greater compacting stresses upon collision with the rim. Lower porosities result. The height of fall of agglomerates is also influenced by the angle of inclination of the disc. For a steeper angle the height of fall increases and should produce more compact agglomerates. This effect is offset, however, by less hold-up and residence time in the pan as the slope increases. Rim height can be used to control porosity in that a greater height leads to a greater amount of material in the pan and thus to increased pressure and residence time and consequently greater compaction.

To obtain a consistent product, inclined discs must be operated uniformly not only mechanically but also with regard to solids and liquid binder properties and feed rates.

2.1.3. Disc dimensions, speed, capacity and power

Available models range from laboratory units 1 ft (0.3 m) diameter to production models greater than 20 ft (6.1 m) diameter. Pietsch [24] has surveyed scale-up for inclined disc agglomerators and offers the following relationships for the approximate design of a unit of diameter, D , in metres:

$$\text{rim height (or pan depth), } H \cong 0.2D \quad (9)$$

$$\text{power consumption, } P = 1.1D^2 \text{ kW} \quad (10)$$

$$\text{throughput, } Q = 1.5D^2 \text{ Mg/h} \quad (11)$$

The rotational speed of inclined discs is normally considered in relation to the critical speed, N_c (min^{-1}), of the agglomerator:

$$N_c = \frac{42.3}{\sqrt{D}} \sqrt{\sin \beta} \quad (12)$$

where β is the angle of inclination to the horizontal and D is the disc diameter in metres. In general, inclined discs are operated in the range $0.5N_c$ to $0.75N_c$.

Most manufacturers quote dimensional, capacity and installed power data for their range of disc agglomerators. One such set of data is given in Table

TABLE 3.2
Characteristics of a range of inclined discs. (Courtesy Feeco International, Inc.)

Diameter (ft)	Depth (in.)	Motor (hp)	rpm	Approximate capacity * (tons/h)
16 in.	3.5	0.25	12 to 36 variable	n.a.
3	8	1	9 to 27	0.35
4 ft 6 in.	9	3	6.7 to 20.2	1
6	11	5	8.1 to 16.2	2
8	13	15	7.5 to 15	4.5
10	15.5	25	12.8 fixed	7
12	17.5	40	11.9	12
14	19.5	60	11.3	17
16	22	75	10.7	26
18	24	100	10.4	35
20	26	125	10.0	44
23	30	150	8.0	58
25	30	200	6.0	69

* Approximate capacity based on dry dust 60 lb/ft³. Capacity depends on the type of material and desired end product. Above rates are average for nominal 0.5 in. pellets.

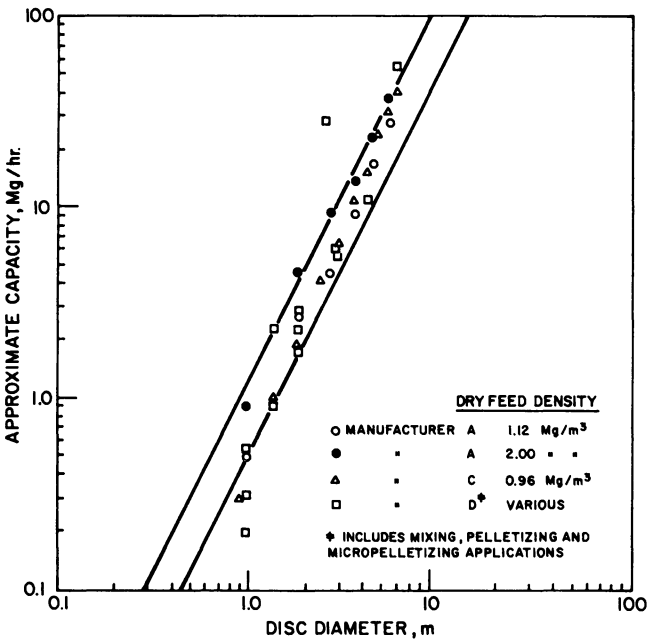


Fig. 3.12. Approximate capacity of inclined discs of various diameters for pellet formation.

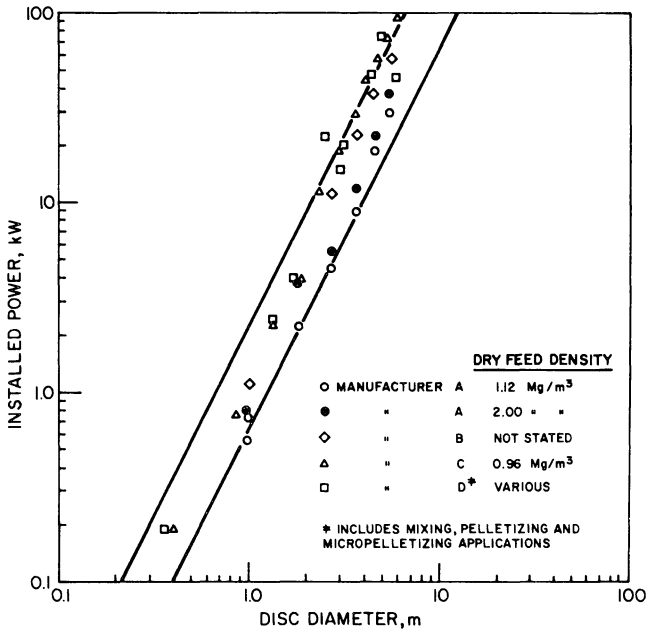


Fig. 3.13. Installed power of inclined discs of various diameters for pellet formation.

3.2. In Fig. 3.12 and 3.13, capacity and installed power, respectively, are shown as a function of diameter for machines offered by several North American manufacturers.

The capacity data in Fig. 3.12 range between $Q = 0.5D^2$ Mg/h and $Q = 1.2D^2$ Mg/h whereas the installed power data in Fig. 3.13 range from $P = 0.6D^2$ kW to $P = 2.3D^2$ kW. These quite broad variations in capacity and power requirements may be explained by variations in rotational speed, pan depth and inclination, feed density and by different requirements in agglomerate properties such as size and porosity. The manufacturers' quoted data are generally more conservative than those quoted by Pietsch [24], which is to be expected.

The above relationships may be used for preliminary design of inclined disc agglomerators. Each application should be analyzed individually, however, with small scale test work in which the behaviour of the material in question can be compared with that of materials whose plant-scale agglomeration characteristics are known.

2.1.4. Modifications of the basic inclined disc

The basic flat disc with rim construction has been modified by various manufacturers. Certain benefits are claimed for each design.

A well-known addition to the basic machine is a separate reroll ring beyond the rim of the main disc (see Fig. 3.14). Formed product-size agglomerates are ejected over the rim into this peripheral ring where they are further smoothed and formed into more spherical shapes. A surface layer of fines, such as coke breeze in iron ore green balls or an anti-caking dust to improve storage properties, may be added in the reroll ring.

Fines and smaller agglomerates can also be separated from the larger agglomerates by the use of multi-stepped sidewalls in the pan (Fig. 3.15). This design is said to lead to stronger, more uniform green pellets since the larger agglomerates, being more positively separated from the fines, roll against the stepped side walls rather than in a soft deep bed of fines. This effect may, however, be counteracted in part by the lower pressure on the larger agglomerates caused by their rolling in a relatively shallow layer.

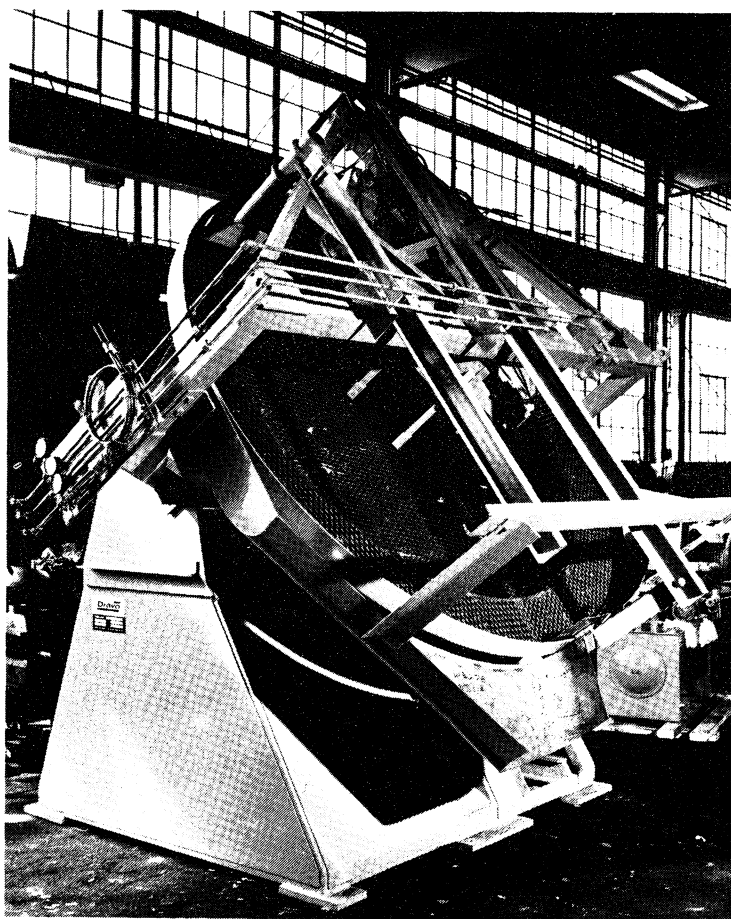


Fig. 3.14. Pelletizing disc with re-roll ring. (Courtesy Dravo Corporation.)

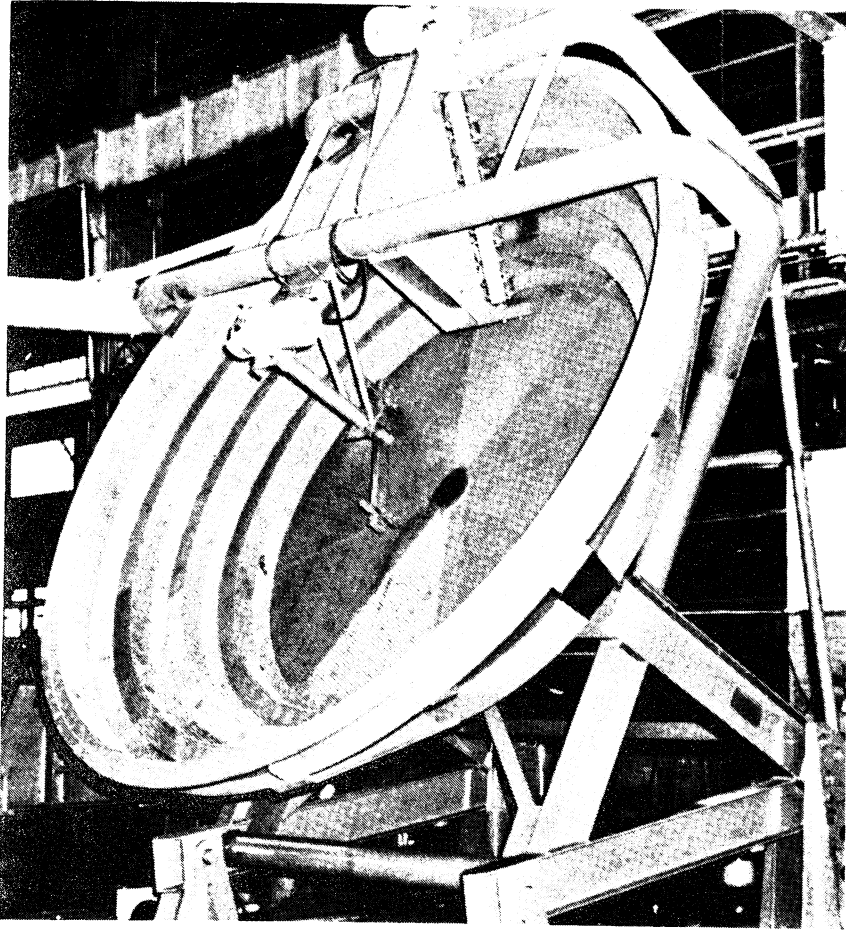


Fig. 3.15. Flying Saucer Balling Disc showing multi-stepped sidewall design. (Courtesy McDowell Wellman Company.)

Equation (9) indicates that normally discs are relatively shallow in depth with a rim height about 20% of the disc diameter. As shown in Table 3.3, however, deeper configurations are also in use in what may be termed “deep pan” or “deep drum” designs (Fig. 3.16). These deep designs increase hold-up in the unit and are said to result in extra firming of the agglomerates from increased bed depth and from the longer retention time for a given throughput. Alternatively, the larger volume of material leads to higher throughputs when retention time is held constant. The larger volume of material in the process makes the unit less susceptible to fluctuations in feed properties and rates.

TABLE 3.3

Inclined pan depth/diameter ratios (H/D) of various commercial units.

Manufacturer	Diameter range	H/D range
Dravo Corporation	1 ft 2 in. to 19 ft 9 in.	0.10–0.17
Feeco International Inc.	16 in. to 20 ft	0.11–0.22
McDowell Wellman Eng. Co.	3 ft 3 in. to 18 ft	0.22–0.26
Maschinenfabrik Gustav Eirich	1 ft 4 in. to 14 ft 9 in.	0.25–0.50
Mars Mineral Corporation	2 ft to 14 ft	0.67–0.75

A unique modification shown in the pelletizer in Fig. 3.16 should also be noted. This unit incorporates an integral screw feeder discharging into the centre rear of the deep pan under the charge which is already in process. This arrangement eliminates free-fall of dry fine feeds, thus preventing dusting, and improves physical and visual access to the front of the unit. Reduced space and power requirements are also claimed for this design.

Finally, an agglomerator design combining features of both the inclined disc and the horizontal drum should be noted. This machine, known as a cone pelletizer, is described in Fig. 3.17 and Table 3.4. As the name implies, the vessel is in the form of a truncated cone and the scraped inner surface has a bowl-like shape as build-up takes place during operation. The cone

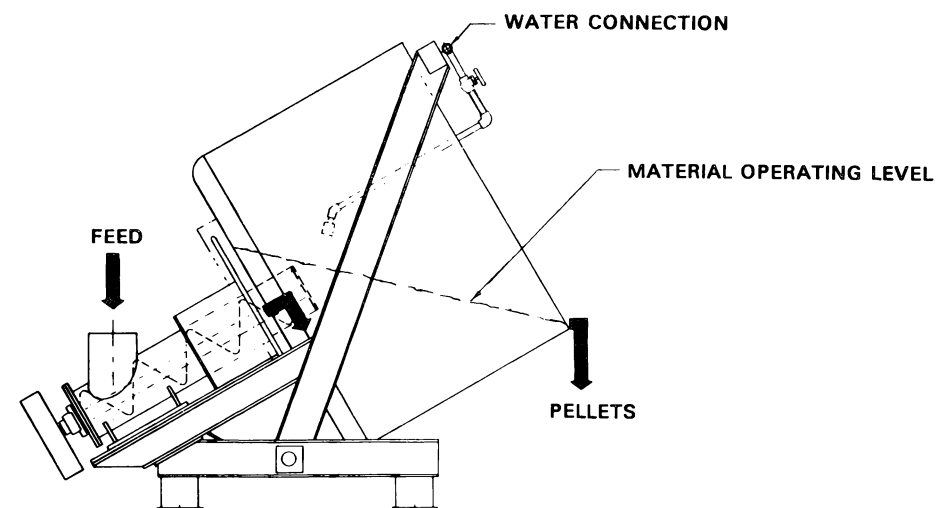


Fig. 3.16. Mars Mineral Pelletizer with deep pan design and rear feed. (Courtesy Mars Mineral Corporation.)

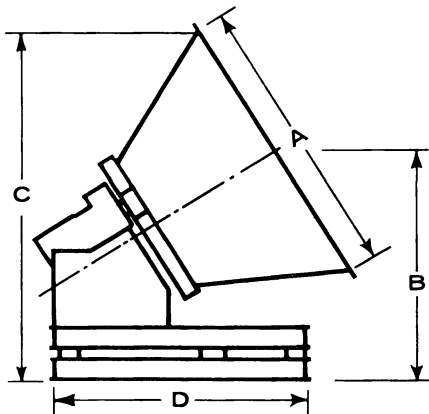


Fig. 3.17. Diagram of Kennedy-Steffensen Cone Pelletizer. (Courtesy Kennedy Van Saun Corporation.)

agglomerator is said to combine the classifying action of the inclined disc at the centre of the vessel with a higher peripheral speed and consequently more vigorous compacting action for the larger pellets as they travel to the base of the cone prior to discharge.

2.2. Drum agglomerators

These machines are widely used in fertilizer granulation and iron ore balling and consist of an inclined rotary cylinder powered by a fixed- or variable-speed drive. Agglomeration occurs when the feed contains an optimal amount of liquid phase and is subjected to the rolling, tumbling action of the

TABLE 3.4
Characteristics of cone pelletizers. (Courtesy Kennedy Van Saun Corporation.)

Diameter (A in Fig. 3.17) (ft)	Approximate capacity * (tons/h)	Motor hp	Typical operating rpm
4	1	5	22.0
8	10	15	18.0
12	35	40	15.0
16	63	75	14.0
18	84	100	12.0
20	98	125	11.5

* Capacities based on iron ore, 150 to 170 lb/ft³ density.

rotating drum. The pitch of the drum, which may be up to 10° from the horizontal, assists material transport down the length of the drum. A retaining ring is often fitted to the feed end to prevent spill-back of feed. A dam ring may also be used at the exit to increase the depth of material and residence time in the drum. In other applications (e.g. iron ore balling), a spiral discharge ring is often used to distribute product to screens downstream of the drum. A trommel (revolving cylindrical) screen integral with the drum may form the discharge end in some cases.

Feed material, consisting of fresh and recycled particles, may be introduced by a belt conveyor, a chute, pipe or similar devices. Liquid phase may be introduced either before or immediately after the solids enter the cylinder. In iron ore balling, premoistened wet filter cake usually constitutes the feed but water sprays may also be located inside the drum for moisture addition to aid balling control. Although water is the usual wetting agent, various solutions and slurries may be used in mixed fertilizer granulation. These may

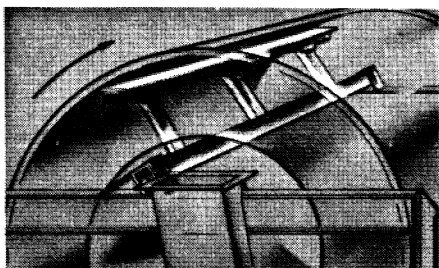
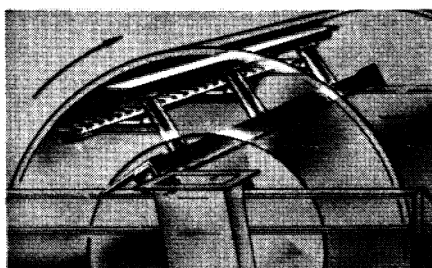
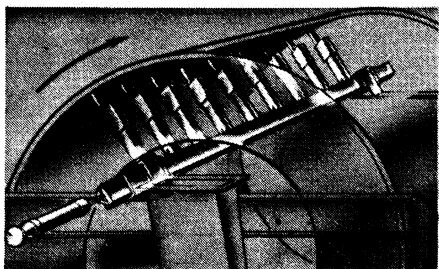
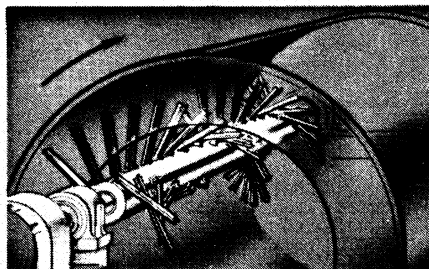
**a****b****c****d**

Fig. 3.18. Internal scrapers for drum agglomerators. (a) single stage adjustable scraper; (b) two stage with tipped cutters and edge-type scraper; (c) powered reciprocating-type scraper; (d) powered rotary spiral with hardened cutters. (Courtesy The A.J. Sackett and Sons Co.)

be introduced as sprays or through other distributors on the bed surface or through spargers under the rolling bed of solids.

As with inclined discs, the character of the internal surface is important in producing the correct tumbling action in the drum. In iron ore balling, the steel drum is commonly lined with a layer of concrete (approx. 1 in. (2.5 cm) thick) or by expanded metal. Various types of internal scrapers, depicted in Fig. 3.18, are used to limit build-up of material on the inside surface and to provide a uniform layer to promote the correct rolling, tumbling action in the drum. Rubber flaps and liners as well as external knockers are used to limit build-up in fertilizer processing.

2.2.1. *Balling circuits and granulation loops*

Because rotary drums do not possess the inherent classifying action of the inclined discs, agglomerates of a wide size distribution are discharged. Drums are therefore operated in closed circuit with screens to recycle the undersized (and crushed oversized, if present) material. In addition, large agglomerates such as green iron ore balls, would require excessively long drums to grow to the required size in one pass. Hence, recycle operation with multiple passes through the drum is adopted. Recycle operation, in addition to requiring more space and equipment additional to that generally used with discs, presents unique control problems.

Consider first the iron ore balling circuit in Fig. 3.19 in which the drum is supplied with damp raw ore at a constant rate together with the undersize returns. In the feed end of the drum, the fresh feed contributes to growth by layering onto existing recycled balls and by the creation of new seeds. For a properly balanced operation, the rate of production of stable new seeds must equal the rate of production of product size agglomerates. This delicate balance is difficult to achieve in practice with the result that a cyclic variation ("surging") in ball production occurs even when the input rate of feed material and its moisture content are held constant [22,26,27]. In typical commercial operation, the surging has a cycle time of 8–12 min and the recycle load may vary from 150% to 300% of the feed rate with the recycle surge amplitude at 20% of this mean value.

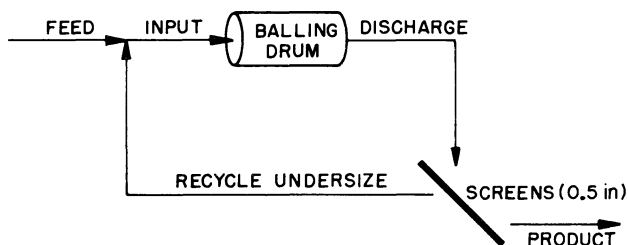


Fig. 3.19. A typical iron balling circuit.

Carter and Wright [26] have described how a surge can develop in iron ore balling. “When an excessively large surge develops in a circuit normally operating well, it is due to a fall-off in growth rate most probably caused by a lowering of moisture level. When this occurs the number of (product) balls passing over the green ball screen starts to fall. The recycle rate increases and . . . seed growth is further inhibited because a greater proportion of new material contributes to the growth of recycling balls. The process tends with decreasing growth rate towards batch operation whereby the recycle balls grow, to the virtual exclusion of seed formation, until they are large enough to pass over the screen. This they do relatively *en masse* and as they leave the balling circuit seed formation is re-established and the cycle is repeated.”

From the above description, it is evident that an increase in growth rate will restore the system to steadier operation. Increasing moisture content by spray additions within the drum is commonly used to reduce recycle and eliminate surging but this may result in the formation of weak, poorly-shaped balls. Thus surging is not only undesirable in that larger recycle capacity is needed, but product quality may also suffer from the unsteady operation. Various schemes are available to reduce the amount of surging. Of primary importance is careful design for consistent operation, since surging can arise not only from variations in feed moisture but also from variations in feed rate and drum speed as well as from changes in the internal drum surface.

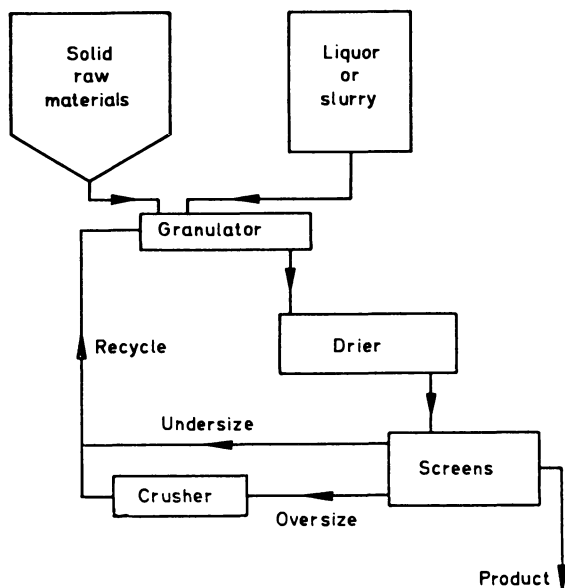


Fig. 3.20. Outline of fertilizer granulation process [8].

Multicircuit installations tend to dampen the effects of surging since the “peak” in production from one circuit may balance a “valley” in production from another. Surging can also be reduced by the use of a two-drum circuit [5] in which the correct proportion of seed nuclei is formed under controlled conditions in the first drum and then passed on to a second where more feed is added to grow the seeds to product size by layering.

A simplified fertilizer granulation loop for drum or mixer agglomeration is shown in Fig. 3.20. A major difference between fertilizer granulation and iron ore balling is the considerably smaller product (about 4–9 mesh in Europe, 6–20 mesh in North America) produced in the former process. This means that oversized as well as undersized material may be produced during a single passage through the drum and must be crushed and recycled together with the undersized product after drying. In addition, these smaller fertilizer granules can be grown predominantly by coalescence (known as “agglomeration” in the fertilizer industry) whereas the layering mechanism is necessary to produce the larger green balls used in iron ore processing (see Section 1).

As with iron ore balling, the primary control of granulation is through the amount of liquid (or solution) phase present. Because of the presence of soluble constituents, the amount of water needed for granulation is roughly established by the chemical composition of the fertilizer. Operational controls are used for minor adjustment of the liquid phase in the granulator to stabilize the process and include adjustment of:

- (1) recycle, including undersize, oversize and product grade material,
- (2) direct water additions, through sprays, steam, etc.,
- (3) temperature in the granulator.

The layering mode of growth is favoured by high recycle levels which provide coarse nuclei for the coating process and by lower levels of liquid phase in the granulation drum due to the relatively small amount of liquid absorbed by the recycled seeds. The coalescence or “agglomeration” mode is favoured by low recycle ratios wherein the drum load is primarily composed of fine, fresh feed material which can readily stick together. Coalescence requires higher liquid phase ratios since the interstices between the coalescing fines throughout the whole granule cross-section must be filled with liquid.

As the fundamental understanding of agglomeration circuits improves and suitable monitoring instrumentation is developed, simulation and automatic computer control [28] of these operations becomes possible.

2.2.2. Drum dimensions, speed, capacity and power

Specifications for some typical drum sizes used in the granulation of fertilizers are given in Table 3.5. The capacities quoted are for guidance only since fertilizer granulation rates depend primarily on the quantity of undersize and crushed oversize material recycled to the drum during continuous operation. The recycle rate varies from less than 1.0 (greater than 50% product size) to 5 or 6 for hard-to-granulate grades. The grade being granulated,

TABLE 3.5

Characteristics of some fertilizer granulation drums. (Courtesy The A.J. Sackett and Sons Co.)

Diameter (ft)	Length (ft)	Installed power (hp)	rpm	Approximate capacity * (tons/h)
5	10	15	10–17	7.5
6	12	25	9–16	10
7	14	30	9–15	20
8	14	60	8–14	25
8	16	75	8–14	40
10	20	150	7–12	50

* Capacity excludes recycle; actual drum throughput may be much higher.

TABLE 3.6

Characteristics of some iron ore balling drums *.

Diameter (ft)	Length (ft)	Drive power (hp)	rpm	Approximate capacity ** (tons/h)	Slope (degrees)	Reciprocating cutter bars	
						Cycles per min	Drive power (hp)
9	31	60	12–14	54	7.1	38	3
10	31	60	12–14	65	8.3	35	3
12	33	75	10	98	7.1	33	3

* Based on information from ref. [33].

** Capacity excludes recycle; actual drum throughput may be much higher.

differing formulations for the same grade, differing plant operations, ambient temperatures and skill all influence capacity in fertilizer granulation [29].

Some typical iron ore balling drums are described in Table 3.6. The L/D ratio of these drums is generally in the range 2.5 to 3.5, somewhat higher than that for fertilizer granulation drums.

Rotational rates, often below 20% of the critical speed * in the past, are averaging 30% or more today and are trending toward 50% for fertilizer granulation [29]. The data in Table 3.6 indicate that iron ore balling drums are operating at around 50% of the critical speed.

* Critical speed for drums given by eqn. (12) with $\sqrt{\sin \beta} \approx 1$.

2.2.3. Modifications of the basic drum agglomerator

Many variations of the rotating cylinder agglomerator are in use or have been suggested. These often involve the use of internal baffles or structures within the drum, as in the multiple-cone drum pelletizer [30] shown in Fig. 3.21. Inside the drum is a series of compartments each separated by an annular baffle. Sidewalls extend from the top of each baffle to the bottom of the baffle next downstream producing a truncated cone shape for each compartment. The inside diameters of the baffles increase from the feed end to the discharge end to give a $\frac{5}{8}$ in./ft (0.05 m/m) slope. One commercial installation has a drum 12 ft. (3.7 m) diameter by 40 ft (12.2 m) in length, contains 17 cones and prepares material for an iron ore sintering strand to produce 4700 tons (4300 Mg) of sinter per day.

The internal structure of the multi-cone drum results in a greater hold-up and longer residence time for material processed. Because of the greater frictional surface area provided by the baffles in contact with the charge, the peripheral speed of the drum is about half that of a normal cylindrical drum. The internals also tend to classify the material, holding back fines at the bottom of a compartment while more fully formed agglomerates ride closer to the charge surface where they pass to the next compartment. An excellent balling action with greater agglomerate compaction is claimed to lead to superior results, for example, higher sintering rates for material prepared for a sintering strand.

Other variations of the rotary drum mixer-agglomerator, fitted with either lifting blades or independent paddle shafts, are also used in feed preparation for sintering operations (see Section 2, Chapter 4).

In the fertilizer industry especially, provision is made for other processing steps, such as drying or chemical reactions, to occur simultaneously with

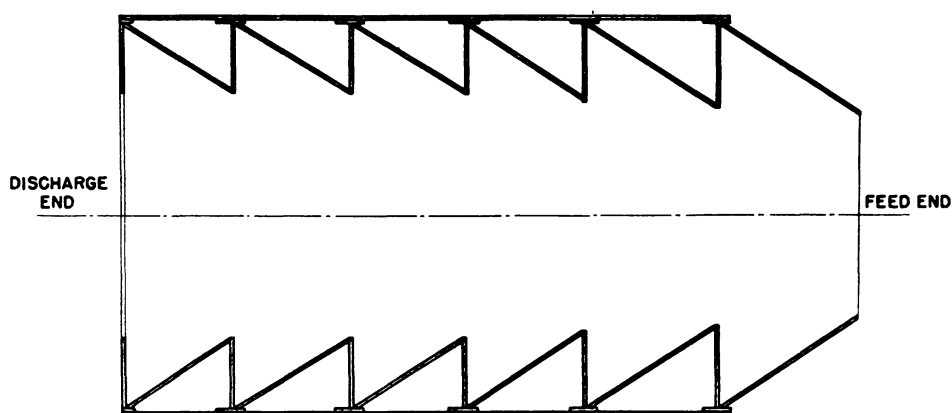


Fig. 3.21. Cross section of multiple-cone drum pelletizer [30].

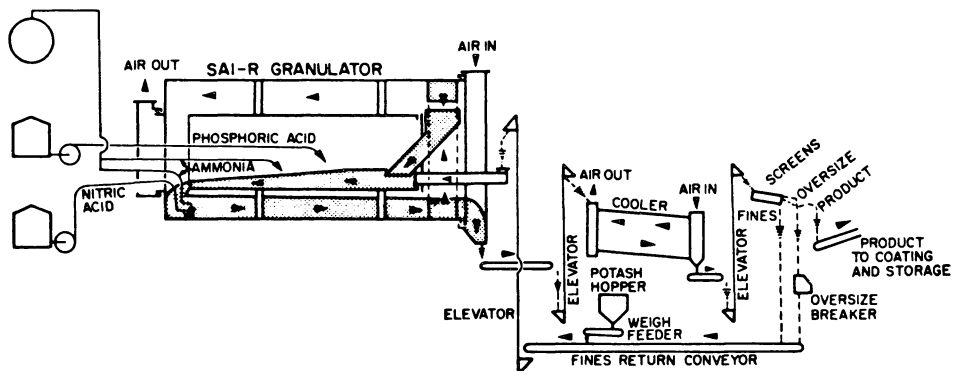


Fig. 3.22. SAI-R granular fertilizer process; reaction, granulation and drying in one vessel [38].

agglomeration within a drum granulator. In the TVA process [31], the heat of neutralization of acids by ammonia is used for drying. An ammoniation-granulation drum may be used in which reactions take place at the feed end of the drum and granulation is continued toward the discharge end. A more complex scheme, the SAI Internal Recycle Process, accomplishes reaction, granulation and drying all in one vessel. The equipment, shown in Fig. 3.22, consists of two drums rotating concentrically one inside the other. Granules are built up, layer by layer, as nitric and phosphoric acids are fed into the rolling, recirculating bed and are immediately neutralized with ammonia. Solid potash particles are also incorporated into the granules as the material recycles. Very high internal recirculation rates can be achieved for layering growth without the need for high capacity external conveying equipment.

2.3. Comparison of drums and inclined discs

The inherent classifying action of inclined discs offers an advantage in applications requiring accurate agglomerate sizing. Other advantages claimed for the inclined disc include less space requirements and lower cost than drums, as well as sensitivity to operating controls and easy observation of the agglomeration process. These latter features lend versatility in agglomerating a wide variety of materials of different degrees of ease of agglomeration.

Advantages claimed for the drum agglomerator compared with the disc are greater capacity, longer residence time for difficult materials and less sensitivity to upsets due to the damping effect of a larger recirculating load. Dusty materials and simultaneous processing steps (chemical reaction or drying during agglomeration) can be handled more easily in a drum.

In a recent quantitative comparison [32] of iron ore balling drum and disc circuits, using the two systems operating in parallel in the same plant with

the same concentrate under the same supervision, some of the main conclusions were:

- (1) Investment costs for the two circuits are about equal.
- (2) The balling disc circuit requires about 10% less building space.
- (3) About equal manual labour is required in each circuit.
- (4) Power consumption is significantly higher in the disc circuit.
- (5) Noise level is considerably lower in a disc circuit.
- (6) A disc circuit requires a smaller product screen.
- (7) Product quality is equal from both circuits.

In the overall cost comparison (items 1–4 above), the balling disc circuit was only about 2% lower in cost than the drum circuit. This difference was considered not to be significant and it was concluded that the choice between the two types of circuits must be made on other factors, including personal choice.

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AGITATION METHODS — MIXER AGGLOMERATION

Virtually all solid-liquid mixers are capable of forming agglomerates and may do so even when not intended. Some of the mixers commonly used intentionally to form agglomerates are described below.

Mixer agglomeration can be broadly classified into two groupings according to the size, density and state of wetting of the agglomerates produced. In the first group are mixers such as horizontal pans and pugmills which are generally intended to produce capillary-state dense agglomerates similar to those made by the tumbling equipment described in Chapter 3. These mixers use agitator/internals within the vessel to provide a positive rubbing and shearing action. Advantages claimed [1] for this method over tumbling methods include harder, stronger agglomerates due to the kneading action, the ability to process plastic sticky materials, and greater tolerance in accommodating variations in operating conditions. Because of the greater compaction achieved, less liquid phase is required for agglomeration in a mixer than in a tumbling device [2]. Disadvantages include generally higher maintenance and power requirements and an irregular product form which may require further shaping, such as in a tumbling dryer.

In the second group of mixer agglomeration techniques, powders are moistened to a much lesser extent than the wet capillary state. Relatively weak powder clusters are formed. An example is the moistening, equilibration, drying and cooling sequence used to produce “instantized” food products.

Specialized equipment has been developed for each of these two types of agglomeration, but some mixers are suitable for both methods. This distinction between granulation-instantizing equipment and equipment to produce larger, more dense agglomerates is somewhat arbitrary, as will be seen in the examples below.

1. Horizontal pans

The horizontal pan mixer was used as a combined mixer-granulator in the early development of fertilizer granulation [3]. A typical machine is depicted in Fig. 4.1. It might be 7.5 ft (2.3 m) in diameter and 18 in. (0.5 m) deep and contain a 0.5 to 1 ton (0.5 to 0.9 Mg) batch of material. Rotation of the pan and mixing blades eccentric to the pan in opposite directions creates a constant state of agitation and, with the addition of plasticizing agent such

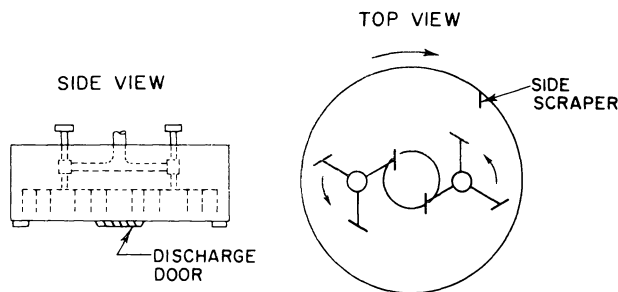


Fig. 4.1. Diagram of horizontal pan mixer for fertilizer granulation [4].

as water, compact agglomerates are formed. With fertilizer formulations, a mixing time of 2 or 3 min is adequate and with an automatic cycle system a production rate of 12 to 15 tons/h (11 to 14 Mg/h) is typical [4] although rates up to 30 tons/h (27 Mg/h) are possible [5]. In modern fertilizer granulation practice these mixers (such as the Eirich intensive counter current horizontal pan mixer) are used to prepare feed going to inclined disc pelletizers [6].

2. Pugmills

These agglomerators, which are also known as blungers, pug mixers and paddle mixers, consist of a horizontal trough(s) containing a mixing shaft(s) (Fig. 4.2). Attached to the shaft are mixing blades of bar, rod, paddle and other designs. The vessel may be of single or double trough design although the latter configuration is most popular. Twin shafts rotate in opposite directions, throwing the materials forward and to the centre as the pitched blades on the shaft pass through the charge. Construction is robust with the body of heavy plate [$\frac{1}{4}$ or $\frac{3}{8}$ in. (6.4 or 9.5 mm) thick] and hardened agitators or tip inserts. Optional features include fume hoods, spray systems and stainless steel construction. In addition to a feed inlet at the end of the mixer, provision may also be made for adding incoming materials at different points along the mixer to ensure that the entire mixing length is used and to add processing versatility.

The general characteristics of the range of pug mixers for fertilizer granulation offered by one manufacturer are given in Table 4.1. Another manufacturer has recently designed and constructed [7] a large double shaft pug mill mixer of stainless steel for handling about 350 tons/h (318 Mg/h) of various fertilizer materials. The unit is approximately 6 ft (1.8 m) wide by 18 ft (5.5 m) long by 5 ft (1.5 m) deep with shafts driven at about 100 rpm by a 400 hp (300 kW) motor. Paddle-type mixers have also been widely used for

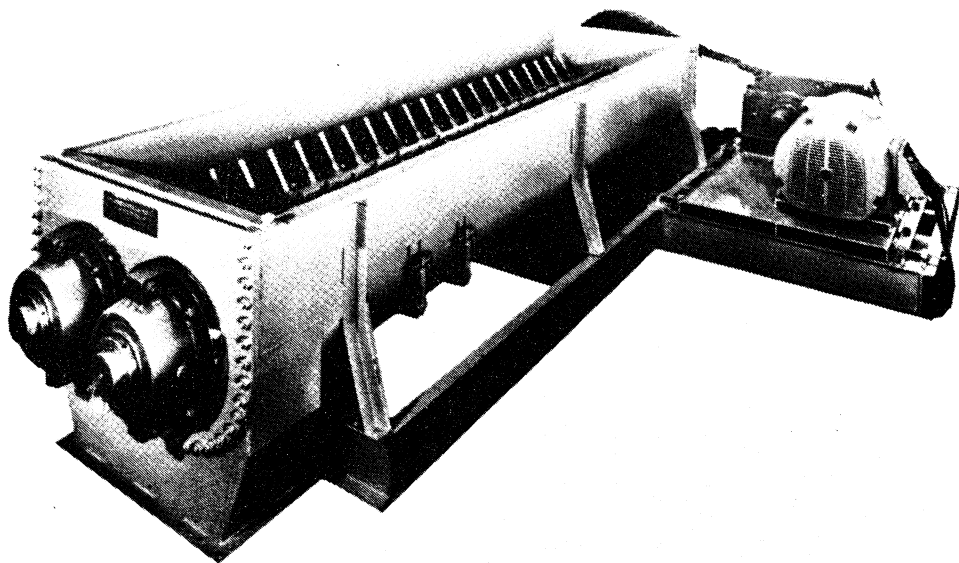


Fig. 4.2. Pugmill or double shaft mixer for fertilizer granulation. (Courtesy Edw. Renneburg and Sons Co.)

TABLE 4.1

Characteristics of pug mixers for fertilizer granulation. (Courtesy Feeco International, Inc.)

Model	Material bulk density (lb/ft ³)	Approximate capacity (tons/h)	Size (width × length) (ft)	Plate thickness (in.)	Shaft diameter (in.)	Speed (rpm)	Drive (hp)
A	25	8	2 × 8	1/4	3	56	15
	50	15	2 × 8	1/4	3	56	20
	75	22	2 × 8	1/4	3	56	25
	100	30	2 × 8	1/4	3	56	30
B	25	30	4 × 8	3/8	4	56	30
	50	60	4 × 8	3/8	4	56	50
	75	90	4 × 8	3/8	4	56	75
	100	120	4 × 8	3/8	5	56	100
C	25	30	4 × 12	3/8	5	56	50
	50	60	4 × 12	3/8	5	56	100
	75	90	4 × 12	3/8	6	56	150
	100	120	4 × 12	3/8	6	56	200
	125	180	4 12	3/8	7	56	300

TABLE 4.2

Drum pug mill used for ferrous sinter mix preparation *. Based on data from ref. [22].

Drum diameter	11 ft
Drum length	14.5 ft
Equipped with counter-rotating paddle shaft	
Paddle assembly diameter	36 in.
Drive power	200 hp
Single motor drives	drum at 8 rpm paddle shaft at 66 rpm
Approximate capacity	300 tons/h

* Total sinter mix preparation carried out in two units, drum-type pug mill followed by second drum to form "seed" pellets.

conditioning (mixing, moistening and micro-agglomerating) sinter strand feed in both the ferrous and non-ferrous metallurgical industries. Pug mills similar to those described in Table 4.1 are still in use in older sinter plants, but more modern plants make use of mixing drums because of their ability to handle larger tonnages with less maintenance and power costs and their ability to handle sticky ores without blockages. One type of mixing drum known as a *drum pug mill* consists of a rotating drum having one internal eccentric paddle shaft. A typical example is given in Table 4.2.

As tonnage requirements have increased, however, the length of paddle shaft mixer required for the necessary residence time has given rise to shaft bearings 40 ft (12 m) or more apart, causing engineering design difficulties [8]. Drums with lifter blades in place of the paddle shaft have therefore been adopted for these larger capacities. Large modern ferrous sintering plants [9] may use mixing drums approximately 16 ft (5 m) diameter by 82 ft (25 m) long operating with the order of 5 min retention time and capacities in excess of 1000 tons/h (907 Mg/h). The objective in sinter feed preparation in such mixers is to eliminate fines (minus 1 mm material) by agglomeration rather than to produce large agglomerates such as those required from the balling drums described in Section 2.2 of Chapter 3.

3. High speed shaft mixers

A more intensive mixing-granulating action than that provided by conventional paddle mixers is obtained in shaft mixers operating at high rotational velocities. These machines are generally single-shaft devices in which the paddles are replaced by a series of pins, pegs or blades. They may be operated

either vertically or horizontally and find application in granulating extreme fines which may be highly aerated when dry and plastic or sticky when wet. The intensive mixing action may achieve agglomeration with short residence times, leading to very compact continuous flow-through designs. Some characteristics of three such machines are given below.

The peg granulator [10] used to agglomerate ceramic clays in the china clay industry is depicted in Fig. 4.3 and some of its characteristics are given in Table 4.3. The pin mixer [11] used to form carbon black pellets is shown in Fig. 4.4 and some of its operating parameters are summarized in Table 4.4. These machines are similar in design and consist of a cylindrical shell within which rotates a shaft carrying a multitude of cylindrical rods ("pegs" or "pins") arranged in a helix. The shaft rotates at speeds of critical importance to machine performance. Wet feed, or dry feed which is immediately moistened, enters the machine at one end and emerges as pellets at the opposite end.

Brociner [10] has described qualitatively the interaction of machine parameters when granulating wet ceramic clay. "The wetter the clay, that is the more plastic it is, the larger will be the granules. The greater the through-put rate, the wider will be the size distribution of the product. The faster the machine speed, the smaller will be the pellets. The greater the work input per unit weight of through-put the better will be the pellet quality, that is to say, the rounder the pellets, and the narrower the size distribution."

The high speed shaft blender-granulator shown in Fig. 4.5 and described in Table 4.5 uses adjustable knives on a free-hanging shaft to produce agglomerates the greater proportion of which are in the 0.5–1.5 mm size range. Residence time of the material in the mixing chamber is usually 0.5–1.0 sec and the device may be described as an "in-line" agglomerator suitable for microagglomeration of a wide range of products. A continuously-deformed neoprene wall is available for sticky materials to overcome build-up. To

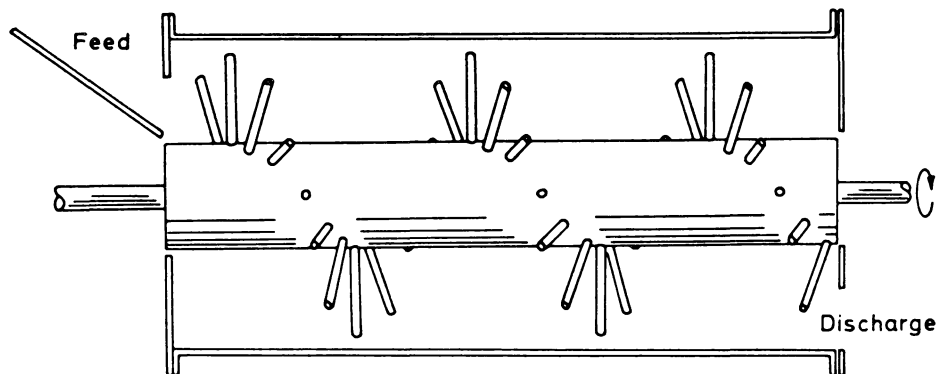


Fig. 4.3. Diagram of a horizontal peg granulator for ceramic clay preparation [10].

TABLE 4.3

Peg granulator used for granulation of ceramic clay. Based on data from ref. [10].

	Laboratory machine	Production machine
Cylindrical casing, diameter	0.75 ft	5 ft
length	3 ft	7.5 ft
Peg-shaft speed	500–1500 rpm	75–225 rpm
Feed material	China clay, about 20% moisture content	
Capacity	0.05 tons/h	15 tons/h
Power consumption	as low as 2 hp · h/ton	

make larger agglomerates, this mixer can be used as a premixer for wetting material going to a balling device. The capacity of a balling disc can be increased in this way.

4. Powder blenders and mixers

In applications such as the preparation of tableting feeds and the manufacture of detergent powders, the aim is to produce small agglomerates (usually 2 mm diameter and less) with improved flow, wetting, dispersing or dissolution properties. This is accomplished by superficially wetting the feed powder, often with less than 5% of bridging liquid in the form of a

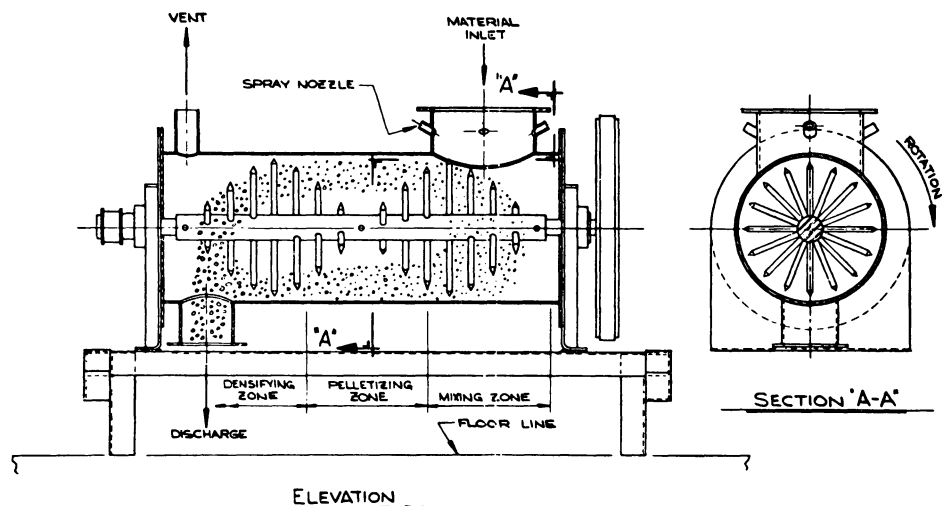


Fig. 4.4. Diagram of a pinmixer for pelleting of carbon black [11].

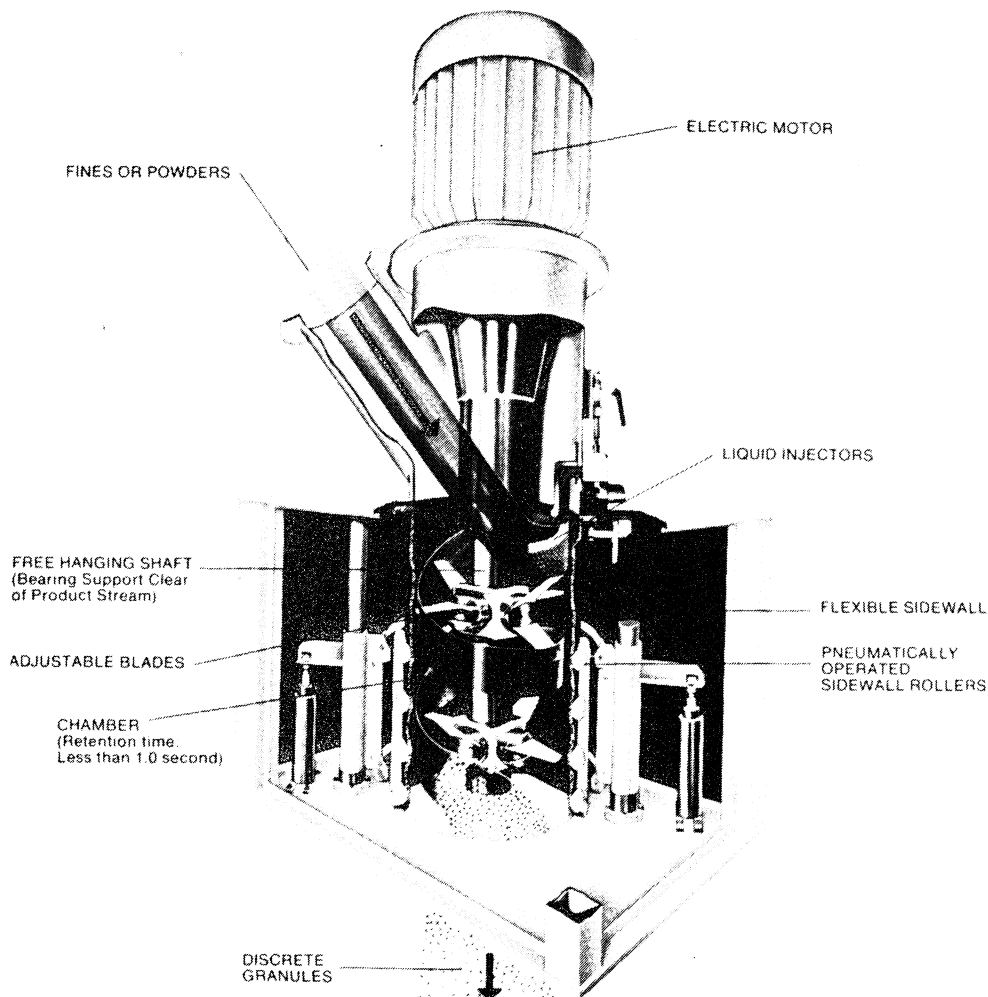


Fig. 4.5. K-G/Shugi vertical continuous blender-agglomerator. (Courtesy of Bepex Corporation.)

spray, steam, mist, etc. The wetting is carried out in a relatively dry state in standard or specialized powder mixers in which the mass becomes moist rather than wet or pasty. Techniques for granulation and instantizing are described in this and the two subsequent sections.

In the standard wet granulating method used to produce tablet feed in the pharmaceutical industry, sigma blade or heavy duty planetary mixers are often employed [12]. These machines may handle 220 or 440 lb (100 or 200 kg) batches and employ 7 to 10 hp (5.2 to 7.5 kW) drives to knead and

TABLE 4.4

Pinmixers used for pelleting carbon black. Test results with a furnace oil carbon black. Reproduced from ref. [11].

Pinmixers tested	Two existing 20" × 100" rubberlined units	Experimental 30" × 100" stainless steel unit (one only)
<i>Carbon black feed</i>		
Rate (lb/day)	40,000 *	58,000 **
Bulk density (lb/ft ³)	3.2	3.2
<i>Pellets produced</i>		
<i>Wet basis</i>		
Production rate (lb/h)	3470 (total for 2 units)	4648
Bulk density (lb/ft ³)	34.9	35.1
Moisture content (%)	52	
Densification ratio	10.9	11.0
<i>Dry basis</i>		
Production rate (lb/h)	1667 (total for 2 units)	2417
Bulk density (lb/ft ³)	23.2	24.6
<i>Binder</i>		
Specific gravity	1.05	1.05
Injection rate (lb/h)	1800 (total for 2 units)	2230
Use ratio, wt of binder to wt of wet pellets	1.08	0.92
<i>Power consumption</i> †		
Rate (kW)	34 (total for 4 motors)	18.5 (1 motor)
Per ton of wet pellets (kW-h)	40.5	15.2 ††
<i>Product quality</i>		
RoTap test (5 min) (%)	1.5 (production avg.)	1.4 (avg. of 45 samples)
Crushing strength (g)	22 (production avg.)	25 (avg. of 73 samples)

* Average from 18-month production history.

** Average from 5-day test, plus subsequent production.

† Ammeter readings.

†† Cold shell.

mass the moistened charge. Mixing times from 15 min to 1 h may be necessary depending on the formulation. The mass is then wet screened or milled, dried and rescreened to the required size which is dictated by the size of tablets to be produced. With hand screening or oscillating granulators, the

TABLE 4.5

Characteristics of K-G/Shugi blender-agglomerators (shown in Fig. 4.5). (Courtesy Bepex Corporation.)

	Smallest unit	Largest unit
Mixing chamber diameter	160 mm	400 mm
Overall machine dimensions		
length	2.0 ft	4.0 ft
width	1.8 ft	2.5 ft
height	3.0 ft	7.2 ft
Drive power	5 hp	50 hp
Approximate capacity	0.5–2.0 tons/h	10–50 tons/h

Notes: 1. Free-hanging shaft is fitted with 2 or 3 knifeholders, 6 blades on each holder.
 2. Admixed liquid is generally in range 10 to 20% of solid feed rate.
 3. Adjustable shaft speeds in range 1000–3500 rpm.
 4. Normally about 20% of chamber volume is occupied by material.

following guide to screen size for this final sizing has been given [12]:

Tablet size	Screen size
up to $\frac{6}{32}$ in. dia.	20 U.S. mesh
$\frac{7}{32}$ to $\frac{9}{32}$ in.	16 U.S. mesh
$\frac{10}{32}$ to $\frac{13}{32}$ in.	14 U.S. mesh
$\frac{14}{32}$ in. and above	12 U.S. mesh

In many instances, the time-consuming wet-milling step may be omitted if the massed material is of an appropriate granular texture after mixing to go directly to drying. This can be accomplished by the use of more intensive and sophisticated mixer-granulators such as the Littleford Lodige unit shown in Fig. 4.6. Mixing takes place in a horizontal cylindrical vessel approximately 50% of which is occupied by the charge. Plow-shaped mixing tools are concentrically arranged on a central shaft revolving at 80–160 rpm. The plows intermingle the powder and drive material into high speed (3600 rpm) choppers which are independently powered and aid in incorporating the granulating liquid. The choppers also control the upper size of lumps and agglomerates formed. The granulation can be discharged directly to drying and is said to compare favourably with that produced by more traditional methods involving wet milling. Dry powder to granulation is claimed to require less than 10 min, much less time than normal methods. Standard mixers with working capacities up to 170 ft³ (4.8 m³) are available. Application of this mixer-granulator in ceramic press feed preparation has been described [13]. A heavy-duty mixer with specially designed plows for feed preparation in iron ore balling is also available.

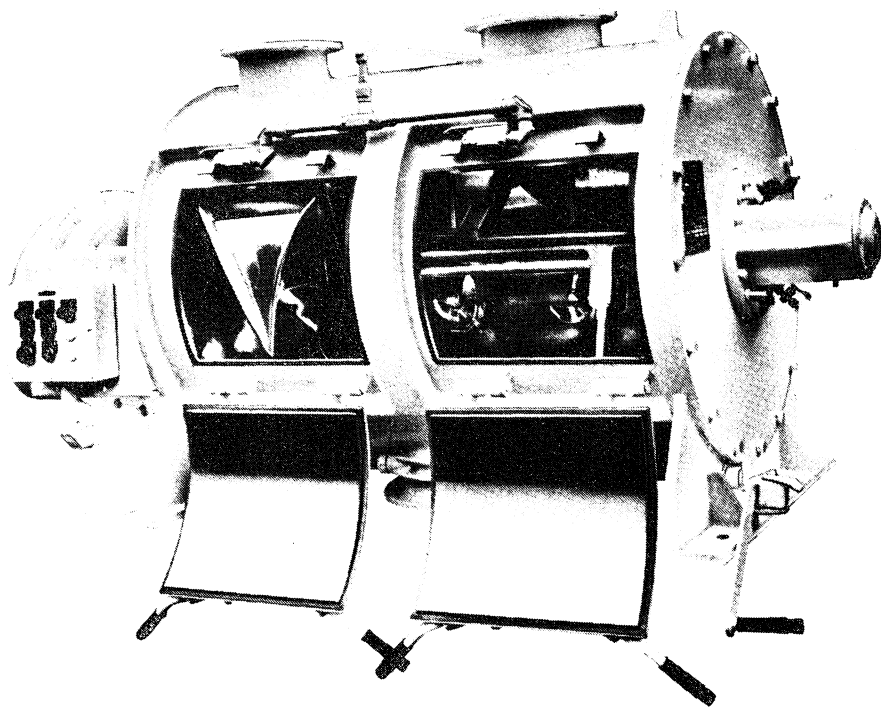


Fig. 4.6. Littleford Lodge mixer/granulator for tablet feed preparation in the pharmaceutical industry. (Courtesy Littleford Bros., Inc.)

5. Falling curtain agglomerators

As the name implies, this technique involves the formation of a falling curtain of powder into which is directed a jet of steam, water spray or mist to wet and agglomerate the product. Small, cluster-type agglomerators are formed.

A novel process to agglomerate home dishwashing detergents by use of a falling curtain agglomerator has been developed [14,15]. Liquid sodium silicate binder is sprayed into a constant thickness falling curtain of ingredients. The curtain is generated in a rotary drum containing an internal cage of bars separated from the drum walls by a spiral ribbon (Fig. 4.7). The cage and ribbon float freely within the drum shell. The cage, together with inertial and centrifugal forces, holds the powder bed against the shell until it falls through the cage under gravity to form a constant density curtain. The spiral serves to recirculate fine material toward the feed end. The curtain of powder absorbs the liquid spray before it can impinge on internal agglomerator surfaces and the free-floating action of the internals keeps all surfaces free of

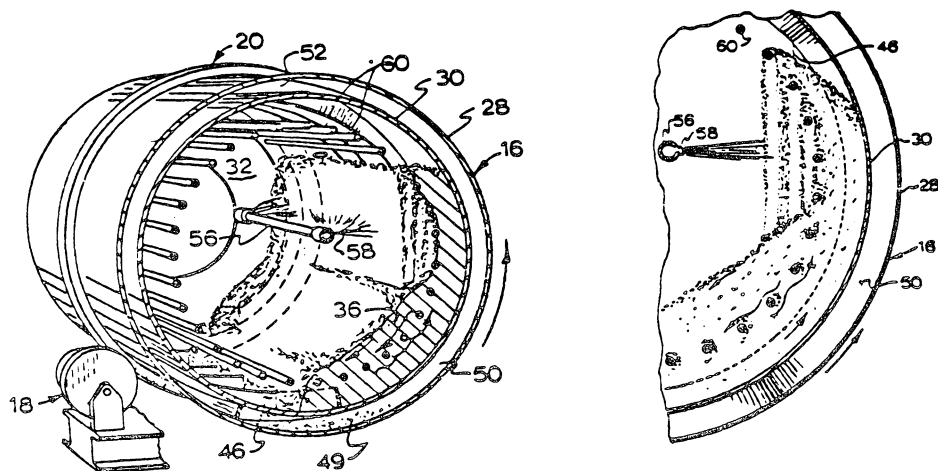


Fig. 4.7. Schematic diagram of internal configuration of constant density falling curtain agglomerator [15].

build-up, both of which prevent lump formation and yield uniform agglomeration. A system to produce 10,000 pounds (4.5 Mg) of dishwashing detergent per hour uses an agglomerator 5 ft (1.5 m) in diameter by 16 ft. (4.9 m) long. Residence time is at least 15 min in the agglomerator followed by 30 min in a conditioning drum to allow surface moisture to equilibrate within the agglomerates. Ambient air is circulated through both units to aid in drying the binders. A product size distribution with less than 1% coarser than 8 mesh and 9% finer than 100 mesh results from normal operation.

Powders for cluster-type agglomeration may also be formed into a falling curtain by various arrangements of feeder boxes and vibratory distributors [16,17]. For example, instantized coffee agglomerates can be made by forming fine coffee powder into a falling curtain which is then carried horizontally by jets of steam directed through the curtain. This steam entrainment is carried out over a conveyor which transports the agglomerates to a drying stage. The success of the agglomeration is said to depend on maintaining a suitably moist atmosphere over the conditioning conveyor.

6. Continuous flow mixing systems

These systems are commonly used in the agglomeration of powdered food products. Fine particles are contacted, after their surfaces are made sticky in a controlled manner, to form clusters. After drying and cooling, the clusters have a coarse, porous structure which improves the wettability, dispersibility

TABLE 4.6
Food items produced by cluster agglomeration [20].

Coffee	Milk/starch/flour	Banana/sugar
Coffee substitute	Cocoa/sugar	Strawberry/sugar/milk
Skim milk	Cocoa/sugar/milk	Instant starch
Whole milk	Beverage whiteners	Ice cream mix
Filled milk	Milk/sugar	Alginates
Baby food	Pineapple/sugar	Whey (precrySTALLIZED)

and solubility of powdered foods to make them “instant”. Food products agglomerated in this way are listed in Table 4.6.

Continuous flow mixers bring together separate powder feed and wetting liquid streams in a moistening chamber. These feed streams must, of course, be well mixed prior to reaching the agglomeration zone to yield uniform products. Many different devices, such as rotating cones, powder funnels, vortex tubes, etc. have been described [18–20] in the literature to provide the moistening-agglomerating zone. The falling curtain coffee instantizers mentioned in Section 5 can also be included here.

The Blaw-Knox Instantizer-Agglomerator [21], which is used to cluster-agglomerate a wide range of food products, can be taken as representative of

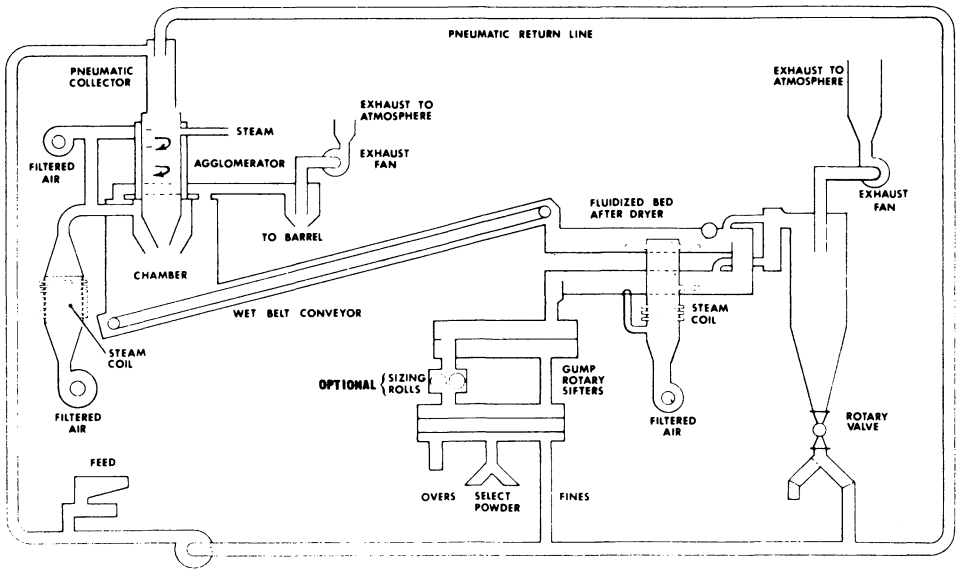


Fig. 4.8. Flow diagram of the Blaw-Knog Instantizer-Agglomerator. (Courtesy Blaw-Knox Food and Chemical Equipment, Inc.)

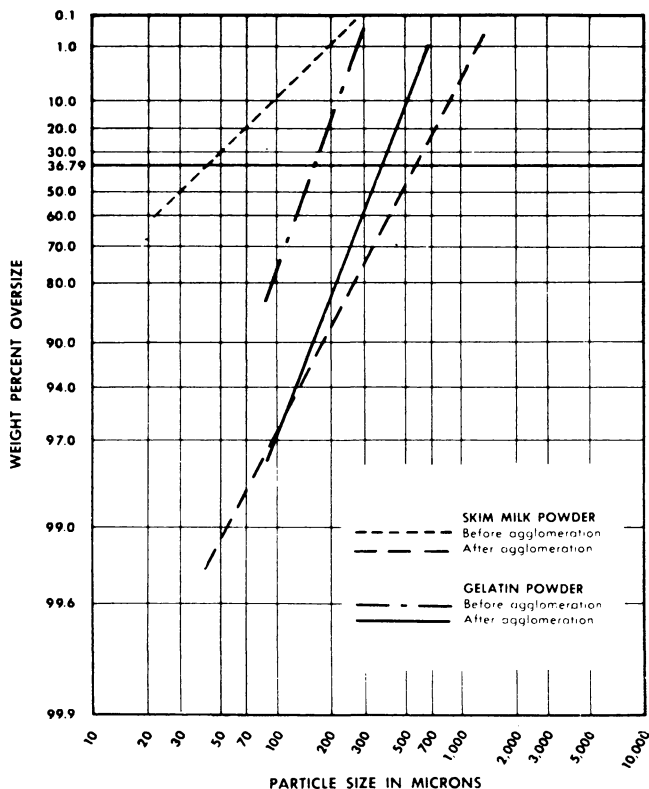


Fig. 4.9. Typical increase in particle size obtained with Blaw-Knox Instantizer-Agglomerator for two food products. (Courtesy Blaw-Knox Food and Chemical Equipment, Inc.)

these systems. A flow diagram is given in Fig. 4.8 while the increase in particle size obtained with the system for two food products is shown in Fig. 4.9. In Fig. 4.8 the feed powder is introduced to the moistening zone by means of a pneumatic conveyor and a rotary valve. The dry powder falls in a narrow stream between two jet tubes which inject the agglomerating fluid in a highly dispersed state. Steam, water or solvents, or a combination of these are used. In addition, air at ambient temperature is introduced through radial wall slots in the moistening chamber to induce a vortex motion. Control of this air flow controls the flow pattern and particle temperature. The reduced temperature serves to condense fluid onto the particles while the vortexing motion induces particle-particle collisions. The clustered material then drops through an air heated chamber onto a conditioning-conveyor where it is allowed sufficient time to reach a uniform moisture distribution. The material then passes to an afterdryer, cooler and sifter followed by bagging of the selected product.

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Chapter 5

PRESSURE METHODS

In these methods of size enlargement, powders are densified and compacted by application of external force in a confined space. Forces involved to produce a stable agglomerate include solid bridges, immobile liquid bonds, surface forces and mechanical interlocking. The success of the operation depends in part on the effective utilization and transmission of the applied external force and in part on the physical properties of the particulate material.

Pressure compaction methods can be broadly classified according to the physical system used to apply the compression force. At one end of the spectrum are punch and die assemblies and molding presses in which particulates are contained in a closed mold and are acted upon by a reciprocating punch or ram. Internal motion and shear of the particles are incidental to consolidation by unidirectional pressing forces. At the opposite end of the spectrum are extrusion systems in which material undergoes definite shear and mixing as it is consolidated while being pressed through an open die or orifice by the action of a screw or a roller. For information on the former die compaction systems used to produce agglomerates of fine detail and uniformity, the reader is referred to a separate monograph on this subject in this series. This chapter will emphasize the high capacity processes which utilize equipment such as roll presses and pellet mills to convert tonnage quantities of fine material into coarser products in which the properties of individual agglomerates are less important.

1. The compaction process

1.1. Mechanism of compaction

With most materials the necessary condition to produce a strong compact is to reduce the porosity to a minimum and increase the density to a maximum. The process of void reduction has been divided into two largely independent probabilistic processes as depicted by Cooper and Eaton [1] in Fig. 5.1. The first is the filling of holes of the same order of size as the original particles. This occurs by particles sliding past one another often accompanied by elastic deformation or even by slight fracture or plastic flow of material. This step is distinguished by the voids being filled by original particles with only

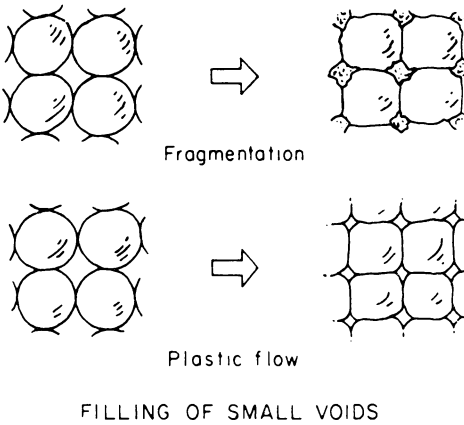
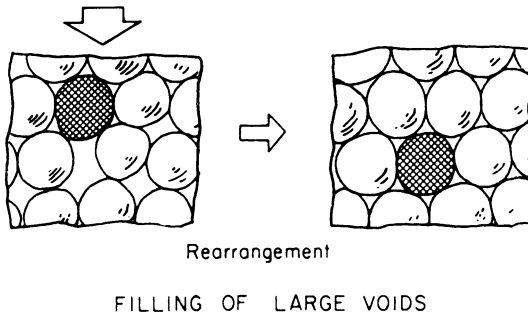


Fig. 5.1. Schematic diagram of different mechanisms of particle compaction [1].

slight size modification by fracture or by plastic deformation. The second process concerns the filling of voids that are substantially smaller than the original particles. These can be filled only by plastic flow or by fragmentation. Plastic flow is more efficient because material is always forced into the voids.

Many equations have been suggested in the literature to represent the compaction of powders in cylindrical die cavities. A selection of these is given in Table 5.1. There does not appear to be a generally acceptable equation to represent all situations. In rotary high capacity equipment, the situation is further complicated by the influence of such factors as scale of equipment (roll or mill diameter) and the configuration of the compacting surfaces.

TABLE 5.1

Equations used to represent compression of powders [24].

Equation	References	Materials used
$\log p = m V_R + b$	Huffine, Ph.D. Thesis, Columbia University, 1953 Stewart, Engineering, 169 (1950) 175, 203	Variety of non-metallic powders Particles of sulfur, sodium chloride, T.N.T.
$\log \frac{1}{1 - \rho_R} = mp + b$	Spencer, Gilmore, and Wiley, J. Appl. Phys., 21 (1950) 527 Heckel, Trans. Met. Soc. A.I.M.E., 221 (1961) 1001	Polystyrene particles Variety of metallic powders
$\frac{V_0 - V}{V_0 - V_s} = a_1 \exp(-k_1/p) + a_2 \exp(-k_2/p)$	Cooper and Eaton, J. Am. Ceram. Soc., 45 (1962) 97	Four ceramic powders
$\log p = m \log \rho_a + b$	Jones, Fundamental Principles of Powder Metallurgy, E. Arnold, London, 1960	Industrial metal powders

Nomenclature:

p = pressure applied to compact
 V = volume of compact at p
 V_0 = volume of compact at zero pressure
 V_s = solid material volume (void-free)
 $V_R = V/V_s$
 ρ_a = apparent or bulk density of compact
 ρ_s = true density of solid material
 $\rho_R = \rho_a/\rho_s$
 m, b, a_1, a_2, k_1, k_2 are constants

1.2. Compaction aids

Lubricants are often employed to facilitate the uniform transmission of forces and reduce undesirable friction effects during compaction. Lubricants may be classified as internal or external types [2]. *Internal lubricants* are materials which are added to the particulate feed not only to improve its flow properties (flow into the mold or die and rearrangement during compaction) but also to assist in the release of the final agglomerate from the mold or die. Internal lubricants are generally used in quantities of $\frac{1}{2}\%$ to 2%. Internal lubricants, especially when used in excess, may interfere with the optimum cohesion of clean surfaces and reduce the strength of the agglomerates produced. *External lubricants* are materials used to prevent friction and wear at

TABLE 5.2
Lubricants for powder compaction [2].

Liquids	Solids
Water	Talc
Lubricating oil	Graphite
Glycerin	Stearic acid
Soluble oil and water	Magnesium stearate
Ethylene glycol	Other metallic stearates
Silicones	Molybdenum disulfide
	Dry starch
	Paraffin, waxes

the die or mold surface and are applied directly to it. A mere trace of an external lubricant may be effective in some cases. Friction at the die surface opposes the transmission of the applied pressure in this region, results in unequal distribution of forces within the compact and hence leads to density and strength maldistribution within the agglomerate [3].

Materials commonly used as lubricants are listed in Table 5.2. An evaluation of lubricants in various applications has been given in Table 2.4 along with information on a number of binders used in tableting.

The role of binder-type additives in developing green and final strength of agglomerates is discussed in Section 4.3 of Chapter 2. It should be recognized that, especially in pressure methods of size enlargement, binders function not only to improve agglomerate strength but also commonly act as lubricants. Optimum agglomeration represents a complex balance of effects due to feed properties (size, size distribution, moisture content), operating temperature, binders and/or lubricants, etc. An experimental program with the type of equipment to be used commercially is generally necessary to determine the best conditions to compact a given material.

2. Equipment

2.1. Piston-type presses

Although die compaction is used most widely in applications (such as tableting in the pharmaceutical industry) requiring strict specifications for the agglomerate, there are a few applications in which piston-type machines are used simply to compress particulates into a larger size suitable for further processing without regard to fine detail.

In the automotive industry and other metal working industries, coarse scrap metal particulates are compressed and recycled to melting operations

through *piston-type briquetting presses* [4–6]. Feed materials are typically cast iron and steel borings and turnings which tend to bond under pressure at least partially by mechanical interlocking. This operation should be distinguished from the much finer metal powders molded into parts such as gears by pressure in the powder metallurgy field.

Piston or ram type briquetting presses are operated as follows. The material to be briquetted is placed into a forming die which is closed on all but one side face. A close fitting ram or piston then enters into the die's open side and exerts the force required to produce the briquet. The briquet is then ejected or "stripped" out of the die. The ram or piston (which is also sometimes referred to as the stamp or punch) which enters into the die can be actuated either mechanically or hydraulically. A typical installation has the following characteristics:

Press type:	reciprocating hydraulic briquetting presses
Die diameter	5 in. (12.7 cm)
Press rating	350 tons (318 Mg)
Oil pressurizing pump	75 hp (56 kW)
Feed material	cast iron and steel borings and turnings
Briquet size	cylinders 5 in. (12.7 cm) dia. × 3 in. (7.6 cm) long
Production rate	3.4 to 3.9 tons/h (3.1 to 3.5 Mg/h)

An alternative high-production means to consolidate large quantities of fines is through the use of *table presses* [7] and standard *concrete block and brick machinery* from the building materials industry [8,9]. Such equipment makes use of vibration and limited ram pressure [e.g. 2000 lb (907 kg) per mold] to consolidate granular material in mold cavities of various sizes and designs. A binder, such as Portland cement, other cements, or various organic binders, is required to develop sufficient strength in the final product after curing. Granular materials such as silicon carbide, iron ore, fluorspar, flue dust, etc., have been treated in this way. Production rates of about 50 tons/h (45 Mg/h) for molding iron-bearing fines are quoted [8].

In the pharmaceutical industry large, often poorly-formed, tablets or "slugs" are made from fine cohesive powders in large capacity *tablet presses* and then comminuted to form a granular feed suitable for final tableting [10]. Slugs are typically 1 or 2 in. (2.5 or 5.1 cm) in diameter and perhaps $\frac{1}{2}$ in. (1.3 cm) thick. Results comparable to those accomplished with this piston and die method are also obtained by roll compaction followed by granulation (see Section 2.2).

Piston-type presses are not suited to large tonnages when a small briquet is required. Their reciprocating nature is a disadvantage since this produces non-uniform loads on the drive motors [11].

Piston-type extrusion presses are described in Section 2.3.1.

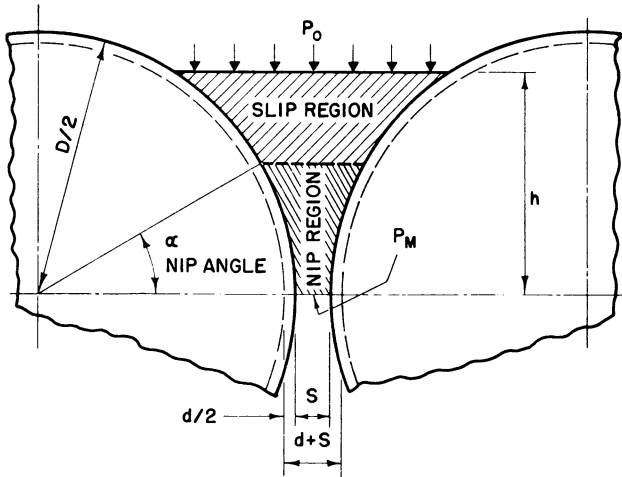


Fig. 5.2. Schematic diagram of a roll compaction and briquetting press [13].

2.2. Roll-type presses

2.2.1. Roll compaction and roll briquetting [12]

In these machines, granular material is carried into the gap between two opposing counter-rotating rolls as shown in Fig. 5.2. As the feed approaches the smallest gap between the rolls, it is pressed into agglomerates. In briquet-

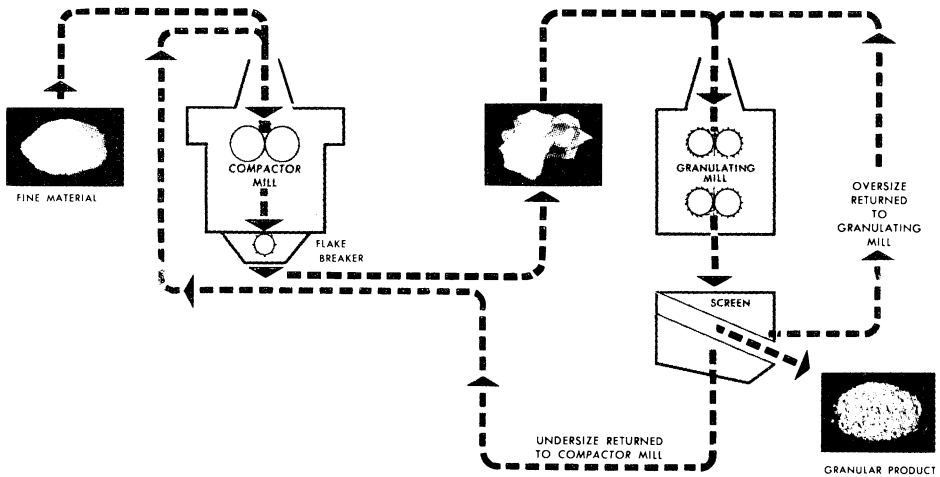


Fig. 5.3. Continuous compaction-granulation process. (Courtesy Allis-Chalmers Corporation.)

TABLE 5.3

Some compacted forms produced with roll-type presses [25].

Sheets	Briquets	Others
Smooth	Pillow	Flakes
Corrugated	Bar	Granules
Dimpled	Ovoidal	Powders
Knobbed	Spheroidal	Slivers
	Tear-drop	

ting machines, agglomerate shape is controlled by means of pockets or indentations on the rolls. In compaction machines, the agglomerated product is in sheet form as produced by smooth or corrugated rolls. The sheet can be granulated into the desired particle size on conventional grinding equipment (see Fig. 5.3). Table 5.3 lists some of the forms of compacts produced with roll-type presses.

Pressure amplification occurs in two regions of a roll press as seen in Fig. 5.2. In the upper region of the roll bite, the material to be compacted is forced into the rolls by the sliding motion between the roll surface and the material. This sliding between the roll and the material occurs above the angle of nip. Below the angle of nip, no slip occurs and the material is squeezed into the final agglomerate. The effectiveness of the rolls in forcing material into the nip reach, the angle of nip, and the pressure amplification depend on the flow properties of the material (see Section 2.2.2).

Roll presses consist of the frame and the two rolls which do the pressing, together with the associated bearings, reduction gear and fixed or variable speed drive. Spacers between bearing housings prevent roll contact and allow adjustment of roll spacing. The frame of the press is designed so that all forces are absorbed internally. The rolls are forced together by means of an hydraulic oil piston. The hydraulic system may incorporate a safety valve which prevents overpressure if foreign material reaches between the roll faces. The rolls consist of a continuous roll shaft, the roll body and attached molding equipment. The molding surface can be either solid or divided into segments. Segmented rolls are preferred for hot briquetting, as the thermal expansion of the equipment can be controlled more easily. Segmented rolls can be made from harder materials more resistant to wear than can one-piece rolls.

A variety of feeding arrangements may be employed to ensure that particulate matter of appropriate bulk density is presented to the nip of the rolls. For smoothly flowing materials gravity feed may be used with a control device to adjust feed rate (Fig. 5.4). For fine powders which tend to bridge or stick and are of low bulk density, some form of forced feed (such as the tapered screw feeder shown in Fig. 5.5) must be used to de-aerate, pre-com-

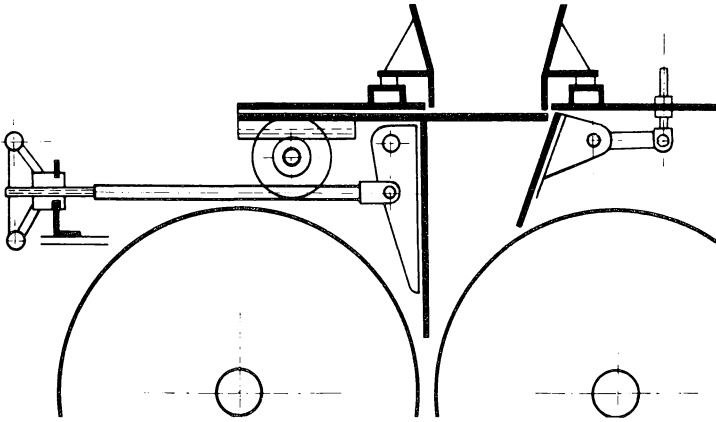


Fig. 5.4. Feed hopper with adjustable tongue for flow control of smoothly-flowing materials to roll presses. (Courtesy Vulcan-Koppert, Inc.)

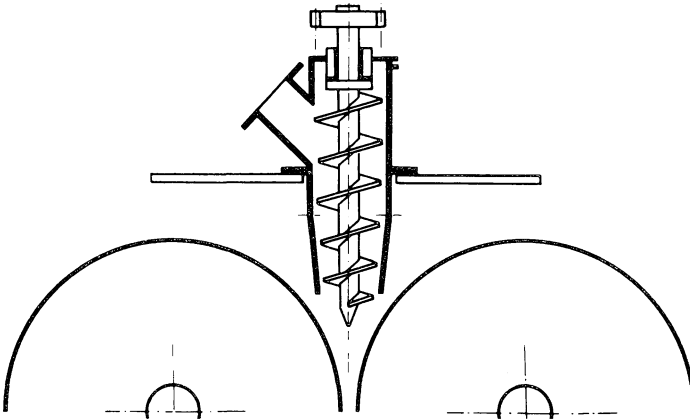


Fig. 5.5. Worm feeder for forced feeding of fine materials of low bulk density to roll presses. (Courtesy Vulcan-Koppert, Inc.)

pact and pressurize the feed into the nip. Automatic control of the feed rate can be accomplished through variable speed motors on these feeders. With control systems to adjust for changes in feed properties, the operating conditions are thus varied to produce uniform products.

2.2.2. Theoretical press design

The design of roll presses for a given application has traditionally been based, in large part, on the experience of press manufacturers gained from many installations. During the last decade, however, a successful theoretical analysis of the operation of roll-type machines has been developed by Johanson [13–16]. Small-scale laboratory measurements of the flow and compres-

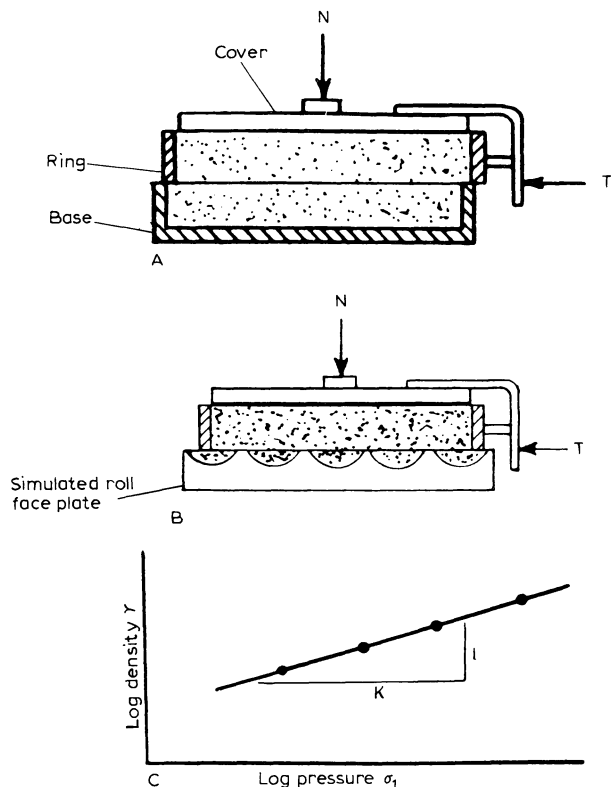


Fig. 5.6. Flow and compression properties of feed solids for theoretical roll press design [15]. (a) Shear cell to measure internal friction of granular solid. (b) Cell to measure angle of friction between roll face and granular solid. (c) Pressure-density relationship of feed material.

sion properties of the feed solids are required as input data. The quantities required are:

(1) The effective angle of internal friction of the powder, δ . This property is a measure of the friction between particles of a bulk solid and can be determined directly in a shear cell (Fig. 5.6a) containing the powder.

(2) The angle of friction, ϕ' , between the roll face and the granular solid. This property is also measured in a shear cell in which the base of the cell is replaced by a sample of the roll surface (Fig. 5.6b). Normal force is applied to the upper part of the cell containing powder and the shear force required to maintain movement between the material and the simulated roll surface is determined. Then, $\tan \phi' = (\text{shear force}/\text{normal force})$.

(3) The compressibility factor, K , of the material. This property is measured in a plunger and die assembly in which the solids are compressed at various pressures [typically in the range 1000 to 70,000 psi (7 to 480 MPa)].

As shown in Fig. 5.6c, K is defined by the slope of the (assumed) logarithmic-linear relationship between pressure and bulk density.

(4) The range of pressures, P_b , over which acceptable briquets can be made with the material in question. These pressures are determined in a plunger and die press at the shortest possible duration achievable.

For further details on these measurements of powder properties, the reader is referred to the original papers mentioned above.

Information is also required on product size and on the press operating conditions, including:

(1) Average briquet thickness, d , for zero roll gap or spacing. This is determined as twice the average pocket depth.

(2) Roll gap, S . A typical minimum value is 0.05 in. (1.3 mm).

TABLE 5.4

Equations derived by Johanson [13–16] for calculation of roll press characteristics.

$$D = \frac{d + S \left(\frac{P_b}{R_1 P_0} \frac{1 + \sin \delta}{1 - \sin \delta} \right)^{1/K} - d - S \cos \alpha}{(1 - \cos \alpha) \cos \alpha} \quad (1)$$

$$RF = \frac{1}{2} P_b WD (F + L/D) \quad (2)$$

$$t = F/2\pi N \quad (3)$$

$$RP = 2.796 \times 10^{-3} P_b WD^2 TN \quad (4)$$

Nomenclature:

- d = average briquet thickness for zero roll gap (in.)
- D = roll diameter (in.)
- f = recycle fraction; fraction of feed material lost from roll bite
- F = roll force factor
- K = compressibility constant for granular solid
- L = length of briquet in direction of rolling (in.)
- N = angular speed of rolls (rpm)
- P_b = maximum pressure to produce a good briquet (psi)
- P_0 = feed pressure (psi)
- R_1 = pressure ratio
- RF = roll separating force (pounds)
- RP = roll power (W)
- S = roll spacing or gap (in.)
- t = pressing time in roll press (min)
- T = torque factor
- W = width of roll (in.)
- α = nip angle (degrees)
- δ = effective angle of internal friction of feed solids (degrees)
- ϕ' = angle of friction between roll face and granular solids (degrees)

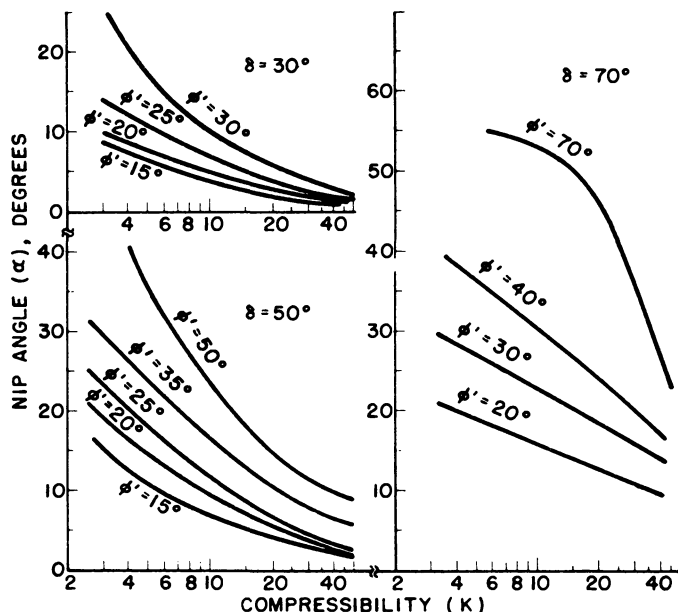


Fig. 5.7. Johanson's theoretical roll press design; angle of nip as a function of material flow properties [13].

(3) Feed pressure, P_0 , applied to the top of the roll bite. A typical value is 10 psi (69 kPa) for screw feed, but a wide range is possible depending on the feeding mechanism.

(4) An estimate of the fraction, f , of material lost from the nip region. Typically f is in the range 0.05 to 0.25 depending on machine design and wear.

According to Johanson's theoretical analysis, the basic characteristics of a roll press can be calculated by eqn. (1)–(4) in Table 5.4, together with the parameters given in Fig. 5.7–5.10. The procedure may be summarized:

(1) Roll diameter, D , is calculated from eqn. (1) where α and R_1 are determined from Fig. 5.7 and 5.8 for the given δ , ϕ' and K . Usually the maximum pressure exerted by the press is set equal to the maximum required briquetting pressure, P_b , to provide a safety factor in the design. A lower applied pressure can be used in press operation, if desirable. Note in Fig. 5.8 a correction may be necessary to R_1 if the dimensionless roll gap S/D is different from 0.01. Since D is unknown, an iterative procedure is necessary in its determination.

(2) Roll-separating force, RF , is calculated from eqn. (2) where F is determined from Fig. 5.9.

(3) Roll speed N is determined from the ratio of the required production

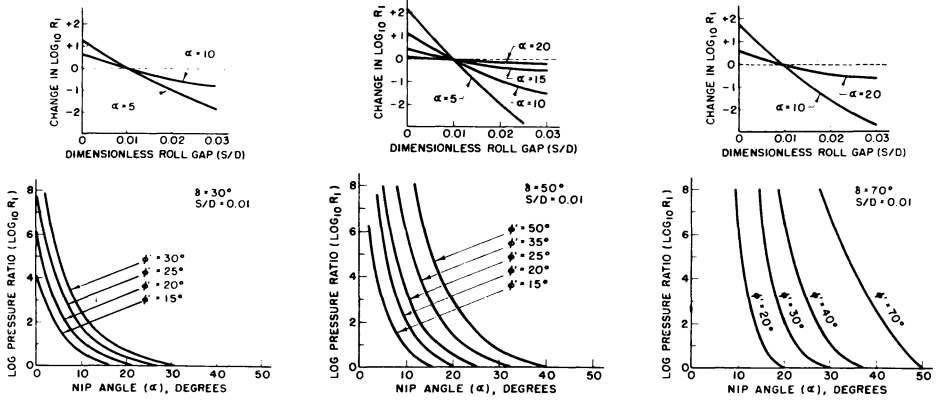


Fig. 5.8. Johanson's theoretical roll press design; pressure ratio R_1 as a function of material flow properties [13].

rate to the weight of briquets produced per revolution of the rolls. Care must be taken, however, to ensure that the feed mechanism can deliver material in excess of that rate and that the minimum time required to make a good briquet is not in excess of the pressing time available on the roll press, given by eqn. (3).

This procedure for determining roll speed is satisfactory for low roll speeds or with very coarse materials. With fine powders and high roll speeds,

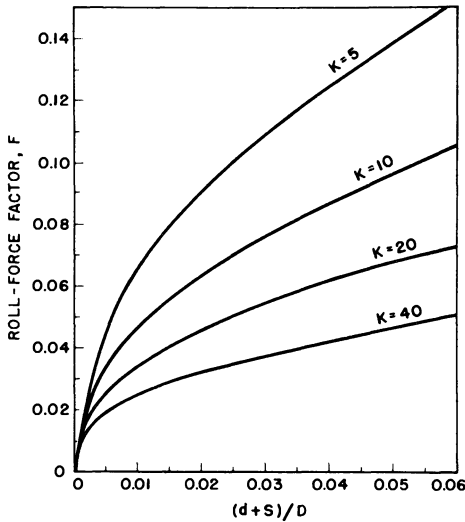


Fig. 5.9. Johanson's theoretical roll press design; roll-force factor (F) versus $(d + S)/D$ [13].

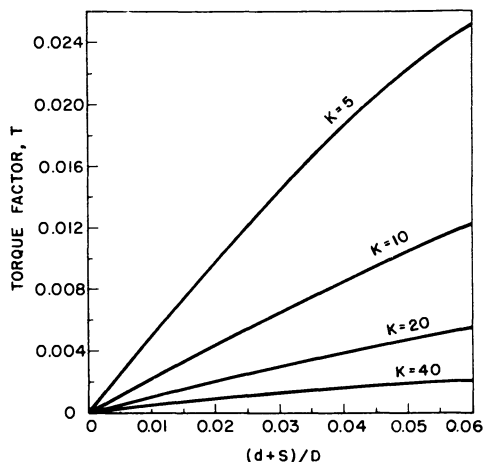


Fig. 5.10. Johanson's theoretical roll press design; torque factor (T) versus $(d + S)/D$ [13].

material permeability and its effect on the escape of liquid and gas from the powder voids must also be considered [16].

(4) Roll powder requirements, RP , are calculated from eqn. (4) where T is determined from Fig. 5.10. This is the theoretical roll power and does not include frictional losses in power transmission.

2.2.3. Performance data

In the previous section, the complex interaction of powder properties and press characteristics which determine such factors as roll force, energy requirements, machine capacity, etc. has been illustrated. Operating experience with a variety of granular materials over a typical range of roll press sizes is presented in Tables 5.5 and 5.6. It must be emphasized that such general information is for preliminary guidance only in the potential capabilities of roll presses. The advice of vendors or consultants must be sought for detailed process design. Manufacturers have laboratory and pilot scale facilities with which to experiment on a given material in order to supply a machine for a specific duty.

2.2.4. Ring roller presses

The working principle of this less well known type of roll press is illustrated in Fig. 5.11 [7,17]. A ring carrying a moulding groove in its inner circumference runs on two driven supporting rollers. A press wheel having a smaller diameter than the ring runs in the moulding groove and acts as the compacting tool. The press wheel is resiliently supported and hydraulically loaded to give the required briquetting pressure generally in the range 30,000 (207 MPa) to 70,000 psi (483 MPa).

TABLE 5.5

Pressure and energy requirements to briquet various materials. (Courtesy Bepex Corporation.)

Pressure	kW-h/ton	Without binder	With binder	Hot
Low (e.g. 500–20,000 lb/in. ²)	2–4	Mixed fertilizers, raw phosphate ore, oil shale, resins	Coal, charcoal, coke, animal feedstuffs	Crude phosphate
Medium (e.g. 20–50 × 10 ³ lb/in. ²)	4–8	Synthetic resins, plastics, PVC, ammonium chloride, DMT (dimethylterephthalate), inorganic salts, animal feed-stuffs	Ferrous alloys (in powder form), fluorspar, nickel powder, molybdenum powder	Iron, potash salts, glass-maker's sand blends
High (e.g. 50–80 × 10 ³ lb/in. ²)	8–16	Metal powders (zinc, vanadium etc.), calcined dolomite, lime, magnesia, magnesium carbonate, sodium and potash compounds, hay and straw	Fly-ash, raw and reduced iron ores	Fly-ash, iron oxides, raw and reduced iron ores, scrap
Very high (>80 × 10 ³ lb/in. ²)	> 16	All types of metal in powder form, metal swarf		Metal swarf

TABLE 5.6

Some typical capacities (tons/h) for a range of roll presses. (Courtesy Bepex Corporation.)

Roll diameter (in.)	10	16	12	10.3	13	20.5	28	36
Maximum roll face width (in.)	3.25	6	4	6	8	13.5	27	10
Roll separating force (tons)	25	50	40	50	75	150	300	360
Carbon								
Coal, coke		2	1		3	6	25	
Charcoal			8			13		
Activated					3	7		
Metals and ores								
Alumina					5	10	28	
Aluminum				2	4	8	20	
Brass, copper	0.5			1.5	3	6	16	
Steel mill waste					5	10		
Iron				3	6	15	40	
Nickel powder					2.5	5.0		
Nickel ore						20	40	
Stainless steel				2	5	10		
Steel								25
Bauxite		1.5				10	20	
Ferro-metals						10		
Chemicals								
Copper sulphate	0.5	1.5		1	3	6	15	
Potassium hydroxide				1	4	8		
Soda ash	0.5				3	6	15	
Urea	0.25					10		
DMT	0.25				2	6		
Minerals								
Potash						20	80	
Salt				2	5	9		
Lime					4	8	15	
Calcium sulphate							13	40
Fluorspar						5	10	28
Magnesium oxide						1.5	5	
Asbestos						1.5	3	
Cement						5		
Glass batch						5	12	

Feed material is fed into the groove ahead of the roller and is pressed into a continuous hoop-shaped strip which may contain indentations caused by projections on the roller surface. The compacted strip is continuously removed from the groove by a stripping tool or finger and breaks into briquets when indented or random pieces when a smooth roller is used.

Ring roller presses with one or two moulding grooves have been described. Capacity is less than that of the more conventional roll presses described in Section 2.2.1, presumably due to the smaller roll width (and hence total

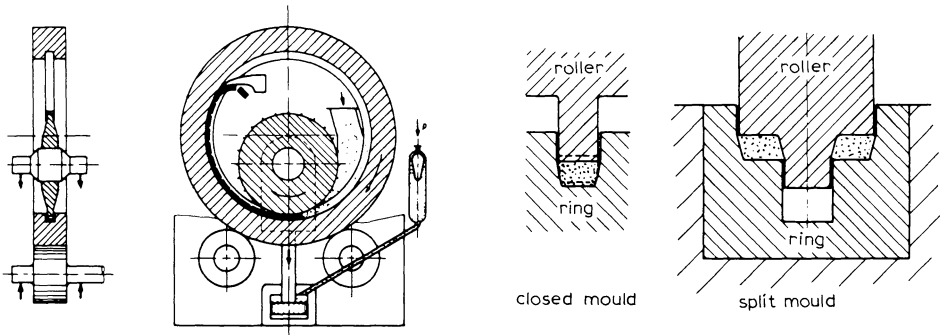


Fig. 5.11. Schematic diagram of the ring roller briquetting press [7].

groove width) available in the developed machines. Characteristics of a larger-size ring roller press are as follows [7]:

Feed material	Compacting pressure	Capacity	Press drive power	Ring speed
Hard coal (hot)	17,000 psi (117 MPa)	3 tons/h (2.7 Mg/h)	35 kW in each case, i.e. 7 to 12 kW-h per ton	3 to 9 rpm
Soda ash	24,000 psi (165 MPa)	3 tons/h (2.7 Mg/h)		3 to 9 rpm
Sponge iron	70,000 psi (483 MPa)	5 tons/h (4.5 Mg/h)		3 to 9 rpm

Ring roller presses differ from more conventional roll presses because of the convex-concave relationship of the ring and roller in the former case, compared with the convex-convex relationship of two rolls in contact in the latter case. This means that pressure build-up and release are both more gradual in the ring roller press. The slower pressure release is claimed to avoid crack formation in the agglomerated product as it expands more slowly after passing the nip of a ring roller press.

Roll-type extrusion presses are described in Section 2.3.2.

2.3. Extrusion presses

This method of size enlargement is distinguished from that in Sections 2.1 and 2.2 in that granular material is compacted and formed by pressing *through* a die or other orifice rather than by pressure *between* two surfaces. Some of the materials being agglomerated by this technique are listed in Table 5.7. Suitable feeds generally include materials which respond to mechanical pressure and some degree of mechanical heat to form into a cohesive,

TABLE 5.7

Some materials being pelleted (roll extrusion).

Adipic acid	Ethyl cellulose	Polyesters
Amides	Feeds	Polyethylene
Antioxidants	Fertilizers	Polypropylene
Asbestos	Fullers earth	Potash
Carbon	Graphite	Rodenticides
Catalysts	Herbicides	Rubber accelerators
Cellulose acetate	Insecticides	Sawdust, sander dust
Chromic acid	Kaolin	Soaps
Charcoal	Latex	Sodium nitrate
Clay	Lignin	Sodium nitrite
Coal and lignite	Methyl cellulose	Superphosphates
Coffee	Nylon	Tobacco
Coke dust	Organic fertilizer	Uranium compounds
Colour concentrate	Paraformaldehyde	Urea
Crumb rubber	Pentaerythritol	Wood pulp and bark
Cryolite	Pharmaceuticals	Xanthates
Detergents	Phenolic resin	Zinc oxide

sticky mass during extrusion. A pre-conditioning step may be involved to accomplish this plastic state, as is the case with brown coal [17] and animal feeds [18]. Development of cohesion and of optimal agglomeration by extrusion depends upon control of:

- (1) particle size,
- (2) temperature,
- (3) moisture content,
- (4) binder and/or lubricants.

Extrusion presses of the piston type, roll type, and screw or auger type are in use.

2.3.1. *Piston-type extrusion presses*

As seen in Fig. 5.12, this type of press works on the principle of a piston-type engine with energy being transmitted from the crankshaft to the piston, rather than vice versa. The press was originally designed to briquet peat and is presently used to compact this material [19] or closely-related feeds such as lignite and brown coal [17] and wood waste [20] (such as sawdust, wood-blocks, shavings and trimmings).

A press consists of two main parts, the driving mechanism and the press head, detailed in Fig. 5.13. The press head contains the mould channel which corresponds to the cylinder in the piston engine analogy. At the rear portion of the press head is a feeding hopper and entrance through which the briquetting material is fed to the mould channel. The material is pushed forward by the stroke of the piston (press stamp) and compacted in a hammer-

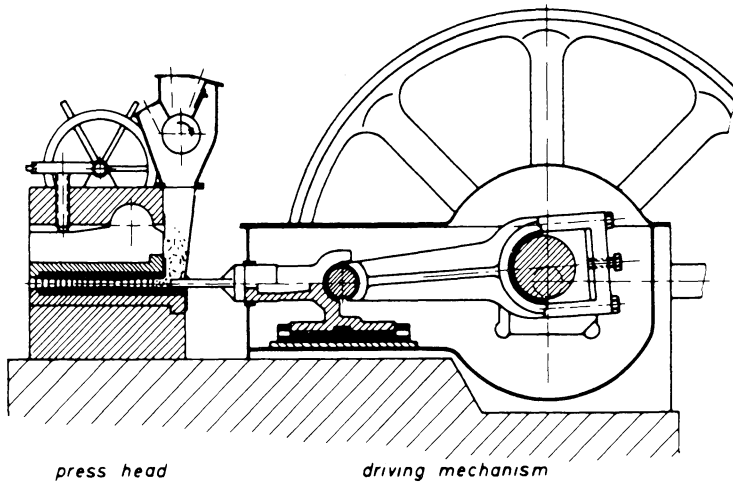


Fig. 5.12. Schematic diagram of the piston-type extrusion briquetting press [7].

like impact against previously-formed briquettes in the mould channel. Typically the mould channel is about 3 ft. (0.91 m) long, 7 to 10 in. (18 to 25 cm) wide, 2.5 in. (6.4 cm) high and contains 15–20 briquets. Elastic re-expansion takes place during the backstroke of the press stamp, and the compacted material in the mould channel takes the form of individual separable briquets rather than a continuous long cylinder. Briquetting pressure typically reaches

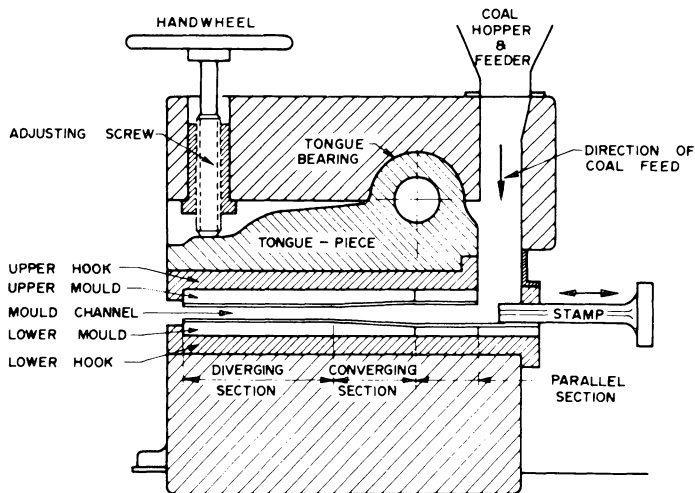


Fig. 5.13. Diagram of the head section of a piston-type extrusion briquetting press [17].

a maximum of the order of 12,000 to 16,000 psi (83 to 110 MPa) applied as an impact over approximately $\frac{1}{12}$ sec [17]. Friction conditions in the mould and hence briquetting pressure are controlled, within limits, by cooling of the briquet mould and by design, especially of the converging section of the mould channel.

Modern piston-type extrusion presses contain multiple stamps and moulds of twin, triple or double-twin construction [7]. At an operating speed of 100 rpm, a triple-type press produces 10.5 tons/h (9.5 Mg/h) and a double-twin type press 16 tons/h (14.5 Mg/h) of lignite briquets. Power requirements are in the range 22 to 35 kW-h/ton (30 to 47.5 hp-h/ton) depending on the feed properties.

2.3.2. Roll-type extrusion presses

These machines, most often known as “pellet mills”, use compaction of material caught in the nip between rolls to force the material in a plastic state through holes in one or both rolls. Adjustable knives shear the rod-like extrudates into pellets of the desired length. Compacting pressure is determined by the resistance of material in the holes. This resistance is a function of hole length divided by hole diameter raised to the third power [21]. Spe-

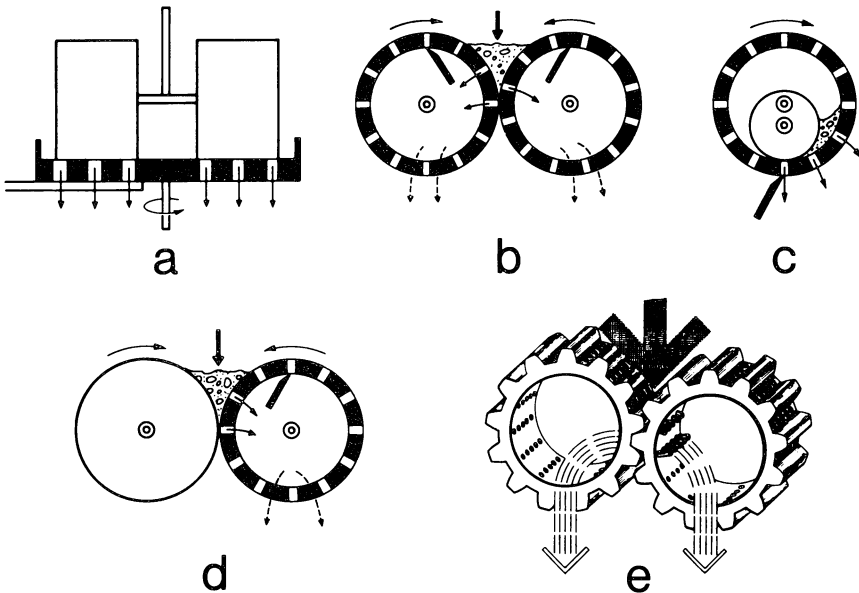


Fig. 5.14. Various designs of roll-type extrusion presses or pellet mills [21]. (a) Horizontal perforated plate die. (b) Extrusion through both side-by-side rolls. (c) Outward extrusion through large die roll. (d) Extrusion through only one of side-by-side rolls. (e) Die holes at root of intermeshing gears.

- 1.
Loose material is fed into pel-
leting chamber.
- 2.
Rotation of die and roller pres-
sure forces material through die,
compressing it into pellets.
- 3.
Adjustable knives cut pellets to
desired length.

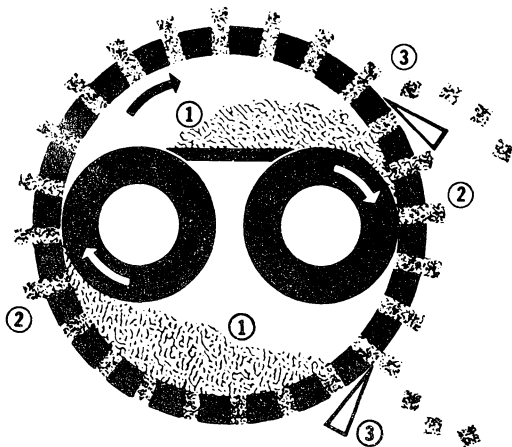


Fig. 5.15. A popular design for pellet mills employing outward extrusion through large die roll. (Courtesy California Pellet Mill Co.)

cial tapers, entrance characteristics and roll die thicknesses are in use to satisfy the pelleting characteristics and product requirements in various applications.

Many designs of machinery which apply the pellet mill principle are available. As shown in Fig. 5.14a, the die may be a horizontal flat perforated plate with rollers running on its upper surface to force material through the plate. Rolls may be either side-by-side with material extruded through one (Fig. 5.14d) or both (Fig. 5.14b) the rolls, or one or more small rolls may be fitted inside a larger die roll, producing outward extrusion (Fig. 5.14c). For specialized applications, two intermeshed gears may be used with material being extruded through die holes located in the gear root (Fig. 5.14e).

The most popular pellet mills employ a ring-type die and two or three rollers mounted in a vertical plane as in Fig. 5.15. Power is applied to the die

TABLE 5.8
Characteristics of pellet mills of design shown in Fig. 5.15.

Horsepower range	10—250
Capacity (lb/hp · h)	75—300
Die characteristics	
Size	up to 26 in. inside diameter × approx. 8 in. wide
Speed range	75—500 rpm
Hole size range	$\frac{1}{16}$ — $1\frac{1}{4}$ in. inside diameter
Rolls	as many as 3 rolls; up to 10 in. diameter

to rotate it around the roller assembly which has a fixed axis. Characteristics of this type of pellet mill are given in Table 5.8. An important aspect of optimum pellet mill operation is the uniform feeding of material over the die surface. This may be accomplished through impellers in the feed chute to accelerate the entering material to the die speed and spread it uniformly. Plows mounted adjacent to the die surface and integral with the roller assembly are also used to provide uniform distribution.

The gear-type pellet mill of Fig. 5.14e is especially useful for difficult applications in which feed material tends to slip away from the nip of smooth rolls [21]. This may occur when the resistance of the die holes is high as with small diameter pellets (e.g. 1 mm diameter). The gear teeth catch the material and force it through the holes; higher compaction ratios and more uniform pressure distribution are claimed.

2.3.3. Screw-type extruders

In this type of equipment, an Archimedean screw is used to pressurize material in a plastic state and force it through a suitable die. This principle is most widely used in molding and forming operations, such as with plastics [22] and structural clay products [9]. These applications are highly specialized and will not be discussed here since they are usually considered to be outside normal size enlargement techniques.

Screw extruders are also used, however, to produce pelleted materials, either as a final product or for further processing, in the food, ceramics, chemical and other industries. Many orifices in a die plate form a multiplicity of extruded rods which are cut into pellets by rotary cutter blades. The extrusion operation is frequently the final step in a sequence of processes which may include binder/lubricant addition, mixing, heating, cooling or vacuum degassing.

In the chemical industry, the formation of catalyst pellets is a typical

TABLE 5.9

Characteristics of pelletizing screw extruders for catalysts. (Courtesy The Bonnot Co.)

Screw diameter (in.)	Drive horsepower	Typical capacity (lb/h)
2.25		60
4	7.5—15	200—600
6	up to 60	600—1500
8	75—100	up to 2000

Notes: 1. Typical feeds are high alumina, kaolin carriers, molecular sieves and gels.

2. Water-cooled worm and barrel, variable speed drive.

3. Die orifices as small as $\frac{1}{16}$ in.

4. Vacuum deairing option available.

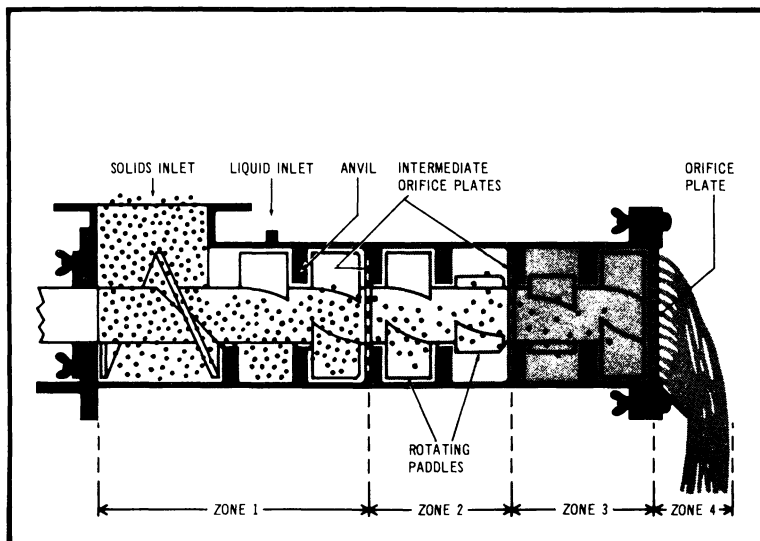
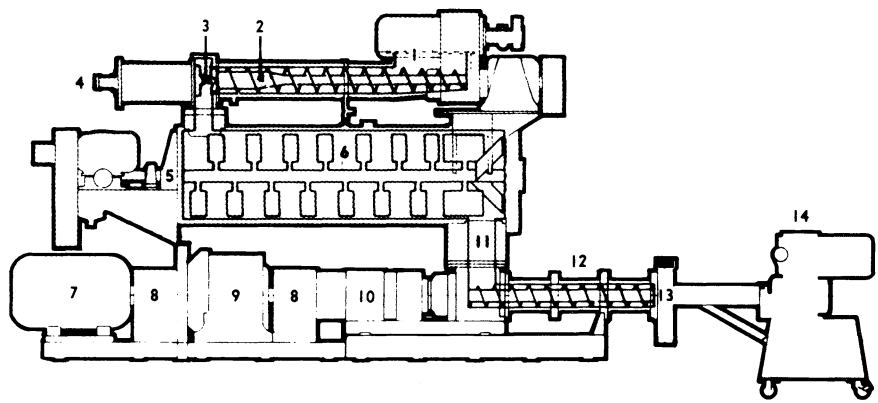


Fig. 5.16. A screw-type extruder used in continuous wet granulation of pharmaceuticals — the Rietz Extruder. (Courtesy Rietz Division, Bepex Corporation.) Zone 1, premixing; zone 2, paste formation; zone 3, intense mixing and dispersal; zone 4, orifice discharge for size and mixing control.

application for screw extruders. The characteristics of a range of machines offered by one vendor for this purpose are given in Table 5.9. Other models for the extrusion of plastic explosives, soaps, minerals, abrasives, asbestos cements, etc. are available with auger diameters up to 18 in. (0.46 m) and capacities up to 60 tons/h (54 Mg/h).

In the pharmaceutical industry, wet granulations of a sufficient scale to justify continuous operation can be carried out in a screw extruder of the design shown in Fig. 5.16. Material is mixed in a number of zones of increasing intensity to form an intimately-mixed plastic mass which is pushed through a perforated end plate. Fixed anvils within the barrel prevent the material from rotating with the screw as it is kneaded and mulled. The spaghetti-like extrusions are dried to form a granular feed for tableting. If the extrusions are dried in a fluidized bed, cylinders 2 or 3 in. (5 or 7.6 cm) in length and $\frac{1}{4}$ to $\frac{1}{2}$ in. (0.6 to 1.3 cm) in diameter are recommended [23] for optimum fluidization.

In the extrusion of snack foods, cereals and pet foods, cooking prior to extrusion is required. This can be accomplished in a steam pressure cooker integral with the extruder such as shown in Fig. 5.17. Screw diameters up to 10 in. (25 cm) are used in this application yielding typical capacities of 4,000 lb/h (1.8 Mg/h) with a 50 hp (37 kW) extruder drive to 12,000 lb/h (5.4 Mg/h) with a 150 hp (112 kW) extruder drive, depending on formulation and product requirements.



**Cooker/Extruder
Components**

- | | |
|------------------------|----------------------|
| 1. Feed Inlet | 6. Cooker |
| 2. Pressure Feed Screw | 7. Extruder Drive |
| 3. Pressure Seal Plate | 8. Coupling |
| 4. Air Cylinder | 9. Gear Reducer |
| 5. Mechanical Seal | 10. Bearing Assembly |
| | 11. Extruder Inlet |
| | 12. Barrel Segment |
| | 13. Cut-off Assembly |
| | 14. Cut-off Drive |

Fig. 5.17. Screw-type extruder with integral cooker for processing of various food products. (Courtesy Sprout Waldron Division, Koppers Co., Inc.)

2.3.4. Screen granulators

These machines produce low pressure compaction and extrusion of moist material by catching it in the nip between a moving bar or wiper and a mesh screen or perforated plate. The granularity of the product depends on the moisture content of the feed. Low moisture produces material of a crumbly

TABLE 5.10

Specifications of a screen granulator used to produce free-flowing feed for tableting in the pharmaceutical industry (Stokes® Model 43-C Granulator; see Fig. 5.19). (Courtesy Sharples-Stokes Division, Pennwalt Corporation.)

Capacity (wet mix through 4 mesh screen)	3000 lb/h *
Screens	Sizes from 2 to 100 mesh
Hopper size	13.75 × 16.25 in.
Motor	1 hp
Floor area	42 × 43 in.
Overall height	51.6 in.

* Dependent on material and desired particle size.

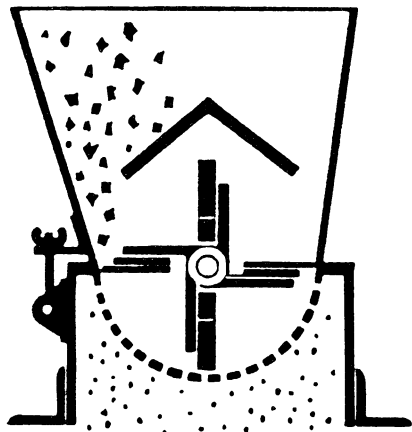


Fig. 5.18. General principle of a screen granulator. (Courtesy Posey Iron Works, Inc.)

texture while higher moisture and plasticity in the feed yields long extrudates.

The general principle of the most common type of machine is depicted in Fig. 5.18. A rotating shaft with attached wipers or bars arranged in cylindrical form oscillates or rotates over a screen or perforated plate of suitable mesh to force material through the screen. A screen granulator used in the pharmaceutical industry to produce free-flowing feed for tableting is pictured in Fig. 5.19. Its characteristics are given in Table 5.10.

The specifications of a larger industrial screen granulator are given in Table 5.11. This machine is designed to sift and granulate mixed bodies of limited moisture content (e.g. 10% moisture) in the ceramics and chemical industry. Its cross section is shown in Fig. 5.18; feed mix falls into a hopper

TABLE 5.11

Characteristics of a screen granulator used in the ceramics and chemical industries (The Lancaster® Pelleter®). (Courtesy Posey Iron Works, Inc.)

	Laboratory model	Standard model	Dual model
Hopper dimensions (top inside)			
length	1 ft. 2 in.	4 ft.	4 ft.
width	1 ft.	2 ft.	4 ft.
Drive motor	0.25 hp	3 hp	2 × 3 hp
“Activator” or wiper construction	nylon brushes, polyurethane wipers		
speed	constant or variable in range 30 to 60 rpm		
Screens	4, 5 and 6 mesh are most common		
Capacity	varies according to mix consistency, screen mesh size and speed		

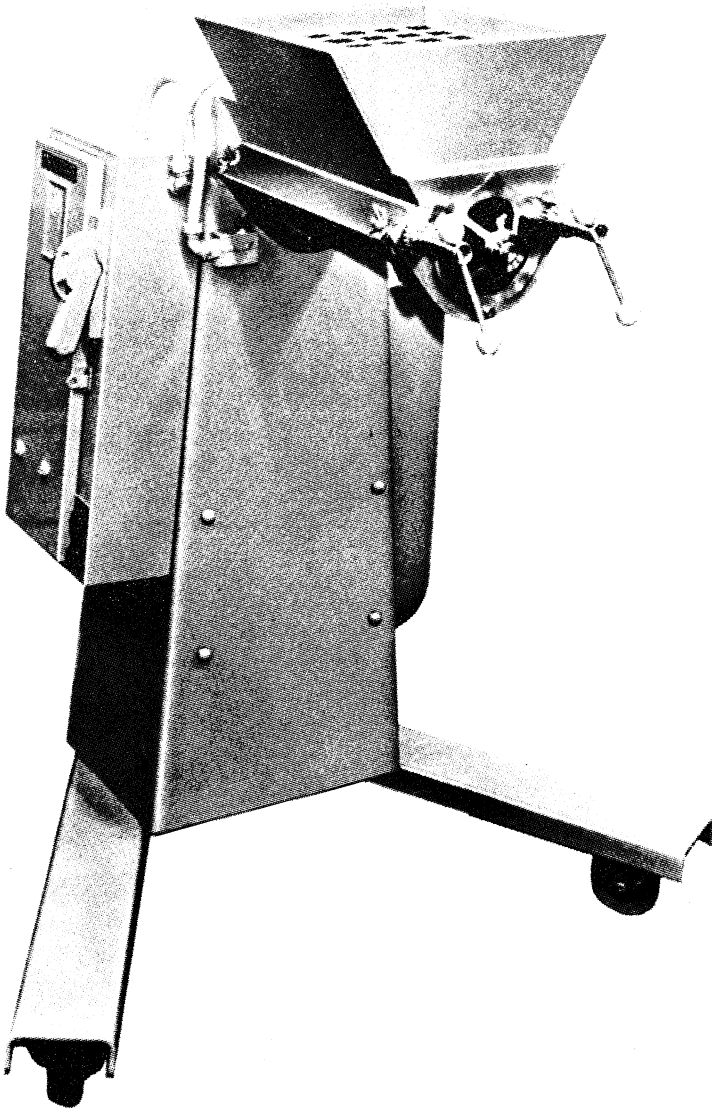


Fig. 5.19. The Stokes Model 43-C Granulator. (Courtesy Pennwalt Corporation.)

where it is directed by a baffle to the “activator”. The activator is a rotating shaft with alternating brushes and wipers contacting a circular half screen to force material through the screen. As noted in Table 5.11, capacity of these machines varies depending on mix consistency, screen mesh size and speed of operation. Capacity is probably of the order of a few tons/h but is best determined by test runs.

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Chapter 6

THERMAL METHODS

The techniques described here depend on heat transfer to form particulates into larger entities. Agglomeration occurs through one or more of the following mechanisms:

- (1) drying of a concentrated slurry or wet mass of fines,
- (2) fusion,
- (3) high temperature chemical reaction,
- (4) solidification and/or crystallization of a melt or concentrated slurry during cooling.

Heat may be transferred directly as in the burning of solid fuel mixed with the particulates in the sintering of ores or indirectly as in the combustion of fuel to produce hot gases in pellet hardening. External heat transfer may also take place across a metallic surface as in drum and belt driers and flakers.

1. Sintering and heat hardening (or induration)

Bonding by treatment at high temperature is often used in conjunction with other agglomeration processes. In the pelletization of iron ore, green balls are indurated (heat hardened) at high temperature. In powder metallurgy, compacts are sintered with or without the addition of binders. The present discussion will emphasize the high tonnage applications of sintering and heat hardening applied in extraction metallurgy to allow fine ores and concentrates to be processed.

1.1. Process mechanism and scope

Sintering, as applied to ore processing, may be defined as the continuous high temperature treatment or burning of thin beds of solids, under induced draft, on a grate. The material to be sintered together with a relatively small amount of solid fuel and water is mixed in various devices to a coarse structured, gas-permeable texture and placed on a grate. Following ignition of the top of the bed, a thin high temperature layer is propagated through the mass in the direction of gas flow. Air cools the product through which the high temperature zone has passed and transfers heat to the material into which the high temperature zone is entering. The added fuel or exothermic bed reactions

compensate for heat losses from the equipment or from endothermic processes during the heat exchange. A cake of hardened material of cellular structure possessing macroscopic pores is thus formed by the fusing together of the feed particles.

Heat hardening is applied to discrete bedded compacts such as green balls or briquettes. In contrast to the sintering operation, induration is accomplished by the external combustion of oil or gas, the products of which are passed through the bed with negligible combustion within the bed. The aim is to harden the agglomerates without fusing them together as is done in the sintering process. By way of summary, a comparison of the sintering and heat hardening processes as applied to iron ore is given in Table 6.1.

TABLE 6.1

Comparison of sintering and pelletizing of iron ore.

	Sintering	Pelletizing
Material treated	Ore fines (finest dust to $\frac{3}{8}$ in. particles); also flue dust, mill scale	Concentrates, 40% to 80% minus 325 mesh
Additives	5% to 9% coke breeze; ground limestone and/or dolomite as fluxing agent	Normally no fluxing agent; 0.5% to 1% bentonite to allow rapid heating and drying and to give greater wet and dry strength
Preconditioning of feed	Mixed with water in pugmill, disc or drum mixer to form coarse-structured nodular bed	Discrete, closely-size green balls formed in disc or drum agglomerators
Thermal consumption	Heat recuperation not usually practised; 1.5–3 million Btu per ton	As low as 400,000 Btu/ton for magnetite. For hematite, about 850,000 Btu/ton. For earthy hematite, up to 1.3 million Btu/ton
Location of operation	Normally at steel works	Normally at mine site
Product characteristics	Large chunks relatively free of fines; 53% to 58% Fe	Uniformly-sized pellets, approx. $\frac{3}{8}$ in. to $\frac{1}{2}$ in. diam.; 62% to 64% Fe
General comments	Recent trend has favoured building of pelletization plants due to chemical and physical uniformity of product. However, because of flexibility of sintering process and its ability to treat many different materials without fine grinding, both types of agglomeration appear to have a definite place in future installations	

Four distinct processes occur during these high temperature agglomeration processes. Each increment of material undergoes:

- (1) drying
- (2) preheating
- (3) firing or high temperature reaction
- (4) cooling.

Ceramic bond formation and grain growth by diffusion are the two prominent reactions for bonding at the high temperatures [2000 to 2500°F (1093 to 1371°C)] employed.

In addition to agglomeration, other useful processes may occur during sintering and heat hardening. For example, carbonates and sulfates may be decomposed or sulfur may be eliminated. Although the major application is in ore beneficiation, other applications, such as the preparation of lightweight aggregate from fly ash and the formation of clinker from cement raw meal are also possible. Nonferrous sinter is produced from oxides and sulfides of manganese, zinc, lead and nickel. An excellent account of the many possible applications is given by Ban et al. [1]. The highest tonnage use at present is in the beneficiation of iron ore and this application will be used to illustrate the process in the following discussion.

1.2. Sintering equipment

The machine most commonly used for sintering iron ores is a travelling grate which is a modification of the Dwight-Lloyd continuous sintering ma-

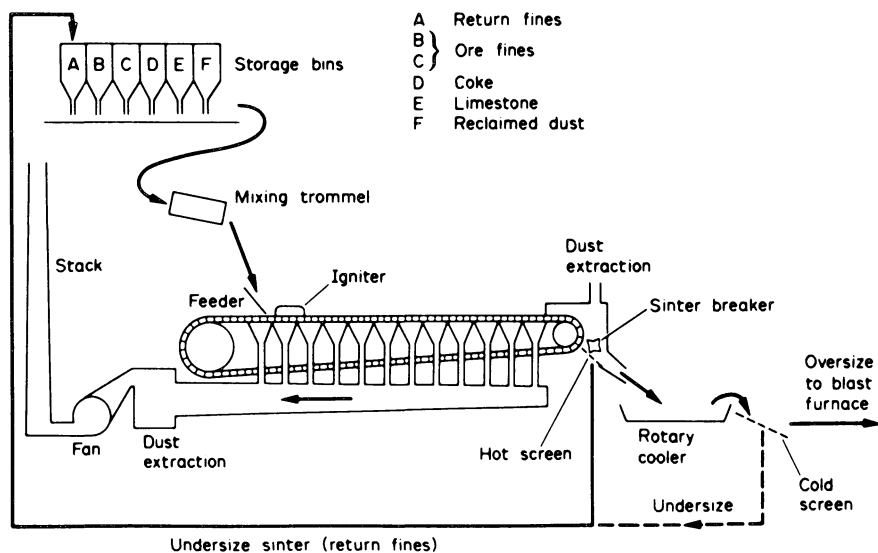


Fig. 6.1. General arrangement of a sinter plant [4].

chine originally used only in the lead and zinc industry. The flows within a typical ferrous sintering plant are shown in Fig. 6.1.

The sintering machine consists of a strong frame of structural steel supporting two gears and steel tracks or guides. Travelling on the track and driven by the gears is an endless system of pallets with perforated bottoms or grates. The train of pallets passes first under a feeding mechanism which deposits a uniform layer of charge, then under an igniter which heats the bed surface to incandescence and finally over a series of wind boxes exhausted by fans. Each wind box is approximately equal in length and width. The speed of the train of pallets and the volume of air drawn through the charge are controlled so that the combustion layer reaches the grate as the pallet passes off the machine.

The sintering machine is a relatively small part of the equipment needed for a complex sintering plant. Auxiliary devices include conveying and storage equipment, mixing and proportioning equipment, fans, dust collectors, etc.

1.2.1. Operating and design information

Details of a number of ferrous sintering machines are found in Table 6.2. A typical modern machine may be 13 ft (4 m) wide by 200 ft (61 m) long with a capacity of 8000 t/day (7200 Mg/day). Included in Table 6.2 are particulars on a large Japanese sinter plant. Such large machines have grate surfaces 16.4 ft (5 m) wide by 394 ft (120 m) long and a production capacity of over 20,000 tons (18,000 Mg) of sinter per day.

Bennett and Lopez [2] have summarized the factors affecting the operation of sinter plants. The capacity of a sintering strand is related directly to the rate at which the burning zone moves downward through the bed. This rate, which is of the order of 1 in. (2.5 cm) per minute, is controlled by the

TABLE 6.2

Ferrous sinter plant performance [2,6].

Type of feed	Hematite ore fines	Fine magnetite conc.	Coarse magnetite conc.	Specular conc. + ore fines	Hematite, magnetite ore fines
Grate area (ft ²)	1293	576	428	1293	6548
Net tons sinter/ft ² /day	3.6	3.6	2.5	3.8	4.3
% Return sinter in mix	30	26	25	35	≈ 28
% Moisture in mix	9.7	9.2	7.0	4.4	5.8
Total million Btu/nt sinter	1.91	1.30	1.67	1.88	—
Bed depth (in.)	15	9	6.8	14	18
Strand speed (fpm)	13.8	9.9	6.8	13	—

- dry balls: compressive strength 9.5 lb (42.3 N)
- heat hardened pellets: compressive strength 500 lb (2.22 kN) or more; tumble test, minimum 95% plus $\frac{1}{4}$ in. (6.3 mm) and no more than 5% minus 28 mesh material.

Several pellet hardening processes are used commercially, and these are summarized below.

1.3.1. Shaft furnaces

Vertical shaft furnaces (see Fig. 6.2) are used to harden natural magnetite pellets. Pellets are fed into the top of the shaft, which is rectangular in cross section, and descend against an upward flow of hot air. Typical operating data are given in Table 6.3.

Balls are charged uniformly over the cross section at the top of a shaft furnace by means of a shuttle belt feeder while the stock level is kept constant by continuously removing hardened pellets from the bottom of the shaft.

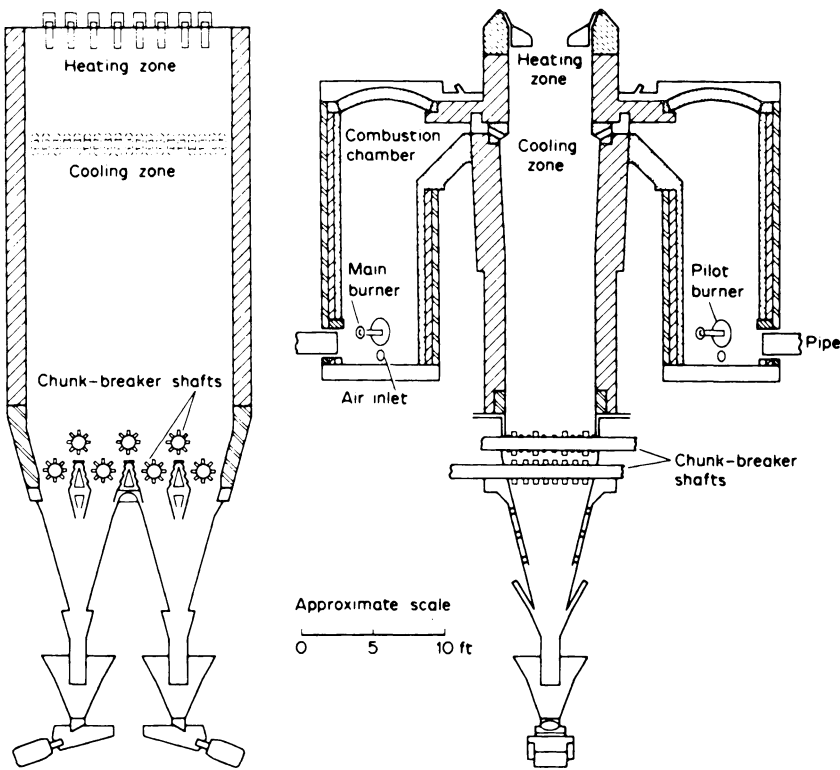


Fig. 6.2. Shaft furnace for hardening iron ore pellets [4].

TABLE 6.3

Typical data on shaft furnaces for iron ore pelletization [2].

Shaft dimensions	7 ft × 14 ft cross section by 45 ft high
Capacity	1000–1200 tons/day
Stock descending rate	1–1.5 in./min
Fuel consumption	400,000–500,000 Btu/ton (magnetite)
Air rate	21,000 cfm at 8 psig
Temperature	
Air from combustion chambers	2350–2375° F
Maximum pellet temperature	2400° F or higher
Gas discharge	400° F
Pellet discharge	700° F

Approximately 25% of the total air is introduced through the combustion chambers and subsequently into the upper part of the column of pellets. Maximum pellet temperature is reached in the top 2 ft (0.6 m) of the stock column.

Shaft furnaces are thermally efficient due to the countercurrent flow of pellets and gas. They are best suited to small tonnage applications and capacities of more than 1200 tons (1100 Mg) per day are not normally built. A shaft furnace is inflexible with regard to changes in the rates of drying and preheating of the pellets and is therefore at a disadvantage compared to travelling grate systems.

1.3.2. Travelling grates

Travelling grate indurating machines were originally developed as adaptations of the conventional downdraft sintering grate. Some characteristics of a modern machine are given in Table 6.4. Generalized process design factors are found in Table 6.5.

Commercial installations differ in air flow arrangements used to accomplish the process functions of drying, firing and cooling. Details of these air-flow arrangements are available [4]. In addition to the common straight-grate machine, a circular-grate design is also in use [5].

Indurating grates differ from sintering grates in the following ways:

(1) Pelletizing grates possess a multiplicity of windboxes divided into major groupings to allow recovery of sensible heat (e.g. air from the cooling section may be used for drying or combustion purposes). This improves fuel efficiency and adds flexibility to the processing steps.

(2) Pellets are held for a long period (relative to that used in sintering) at closely-controlled temperatures to effect hardening.

(3) Pellet cooling is usually done on the same machine used for heat hardening whereas sinter is cooled in separate equipment.

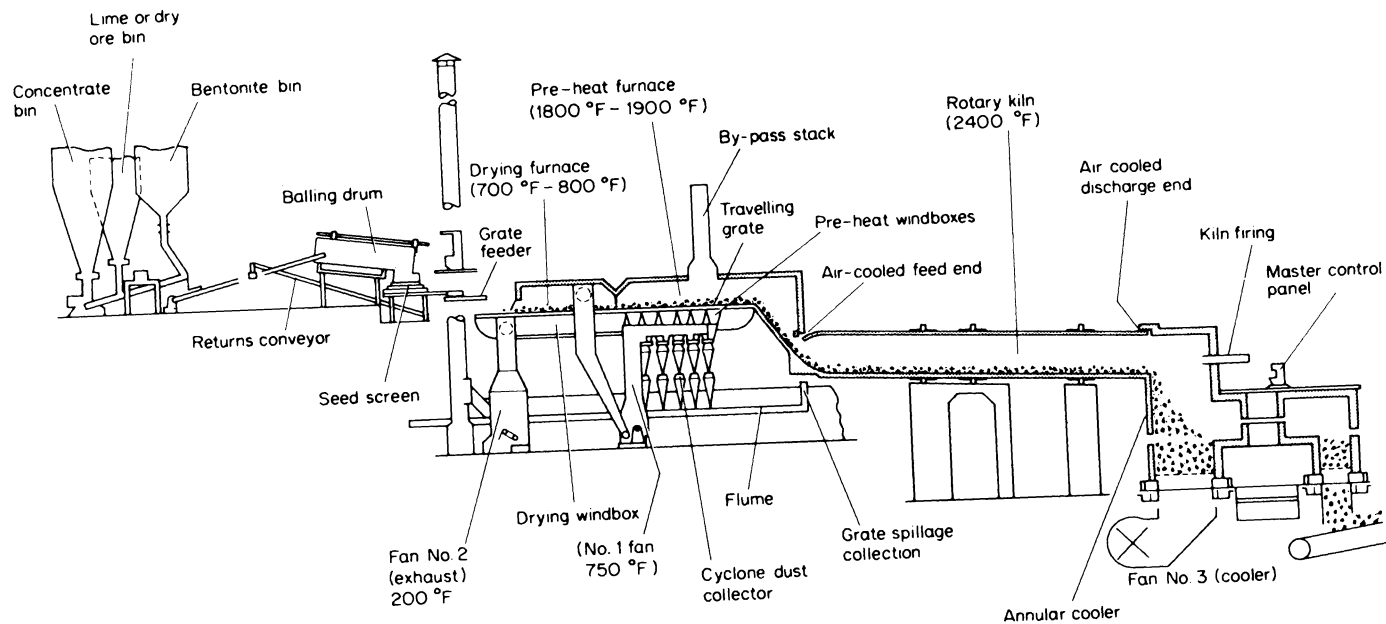


Fig. 6.3. Typical installation of grate-kiln system [4].

TABLE 6.4

Some characteristics of a modern travelling grate iron ore pelletizing machine [7].

Capacity	8650 tons/day
Grate dimensions	11 ft wide × 459 ft long
Windboxes	
number	70
length	6.56 ft
Hearth layer (screened product)	
bottom (thickness)	6 in.
sides (thickness)	4 in.
Initial green ball depth	22 in.
Fuel consumption	1,200,000 Btu/long ton (hematite)

TABLE 6.5

Process design factors * for iron ore pelletizing machines [8]. Factors are based on effective areas.

	Travelling grate	Grate-kiln
Grate loading factor [(long tons/day)/ft ²]	2.1— 2.9	5.1— 5.5
Size factors		
Grate [ft ² /(long ton/h)]	8.2—11.6	4.4— 4.7
Kiln [ft ³ /(long ton/h)]		106 —115
Cooler [ft ² /(long ton/h)]		3.3— 3.6
Air usage ** (lb/lb pellets)		
Kiln		0.9— 1.0
Cooling	2.6— 2.9	1.9— 2.0

* Actual (not design) performance.

** Based on fan ratings.

TABLE 6.6

Some characteristics of a modern grate-kiln iron ore pelletizing machine [7].

Capacity	14,200 tons/day
Grate	
dimensions	18.6 ft wide × 210.7 ft long
windbox number	19
length	10 ft
Kiln	
diameter	25 ft
length	160 ft
slope	0.63 in./ft
Annular cooler	
mean diameter	66 ft
pallet width	10.2 ft
bed depth	2.5 ft

1.3.3. Grate-kiln system

In this process (see Fig. 6.3), pellets are first semi-hardened on a travelling grate where the agglomerates do not move relative to one another. The pellets are then sufficiently strong to be discharged to a rotary kiln where they undergo uniform firing and acquire a denser outer surface than with other firing methods. Countercurrent flow of hot gases and pellets is used. Data on a modern grate-kiln machine is given in Table 6.6, with process design factors included in Table 6.5.

2. Nodulizing

In this process, large somewhat spherical agglomerates are formed from fine particles in a rotary kiln by raising the temperature rapidly to create a liquid phase, which moistens the particles causing them to roll into balls. Subsequent cooling solidifies the liquid phase and hard, fused nodules are produced. The process was used on a limited scale to agglomerate fine taconite concentrates in the United States but seems to have been completely displaced by the heat hardening of previously-formed green balls (pelletizing). Salient features of an iron ore nodulizing-plant are given in Table 6.7. Existing cement kilns were used for nodulizing ores [2] and the “burning” of finely ground feed to form cement clinker might be considered as a further example of size enlargement by fusion similar to nodulizing.

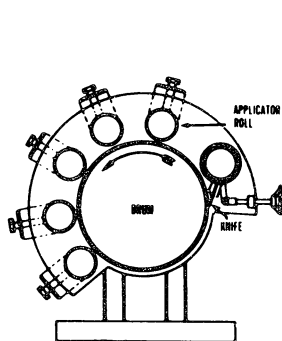
TABLE 6.7
Some features of an iron ore nodulizing plant [2].

Feed	Magnetite taconite concentrate
Feed particle size	90% minus 270 mesh
Nodulizing temperature	2400° F
Kiln exit gas temperature	575° F
Kiln speed	72 rev./h
Limestone addition	82 lb/ton nodules
Nodule production	1274 long tons/day
Fuel consumption (pulverized coal)	1.89 × 10 ⁶ Btu/ton

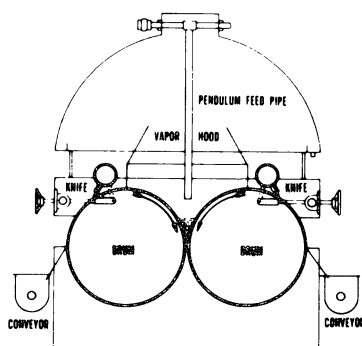
3. Drying and solidification

The drying of solutions and the solidification of melts form an integral part of many of the size enlargement methods discussed in other chapters of this book. These are common mechanisms to harden agglomerates and prepare them for further processing and use. A number of the machines men-

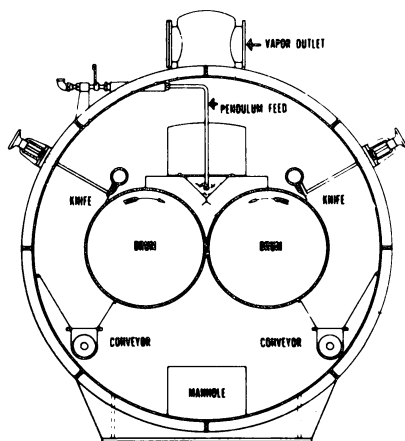
tioned in Chapter 5 for pressure agglomeration are used to preform pastes prior to drying. Soft pastes can be formed into rods by extruders similar to the design in Fig. 5.18 using rollers, bars or wiper blades to force plastic material through perforated plates or grids [9]. Stiffer pastes can be pelleted in machines such as depicted in Fig. 5.14 and 5.15 prior to drying. Various types of dryers, ranging from conveyor bands for gentle action through rotary, fluid bed and pneumatic systems where more vigorous action is acceptable, are used to produce dry granular products from these preforms. Further information on fluid bed and pneumatic (or flash) granulation systems can be found in Chapter 7. A comprehensive treatment of drying of



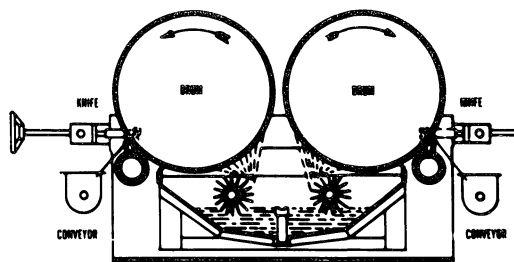
Atmospheric single drum, applicator roll feed.



Atmospheric double drum dryer with vapor hood and pendulum feed.



Vacuum double drum dryer utilizing pendulum feed.



Atmospheric twin drum dryer with splash feed.

Fig. 6.4. Drum dryers illustrating a number of different feed arrangements. (Courtesy Blaw-Knox Food and Chemical Equipment, Inc., Buflovak Division.)

solids in the chemical industry is given by Nonhebel and Moss [10]. Most applications require pilot scale trials to establish evaporation rates in the particular type of preform/dryer combination under consideration.

Drum and belt drying and solidification systems are discussed below. This equipment is capable of forming granular products directly from fluid pastes and melts, without intermediate preforms, by drying or solidification on solid surfaces. Drum and belt systems offer an alternative to the dispersion methods (such as the prilling of sulphur, fertilizers and resins and the spray granulation of clays) described in Chapter 7.

3.1. Drum dryers and flakers

A *drum dryer* (Fig. 6.4) consists of one or more heated metal rolls on which solutions, slurries or pastes are dried in a thin film. The thin film dries in less than one revolution of the slowly revolving rolls and is scraped off by a doctor blade to form a product in flake, chip or granular form. Drum dryers are widely used in chemical processing frequently to dry hydrates, carbonates, arsenates, acetates, phosphates, clays and food products [11].

Some characteristics of drum dryers are given in Table 6.8. In a given application, testing is usually required to establish drum speed, temperature, retention time, product characteristics and feeding equipment. The short time that solid is in contact with hot metal makes these units suitable for heat-sensitive materials. As illustrated in Fig. 6.4, many different feed

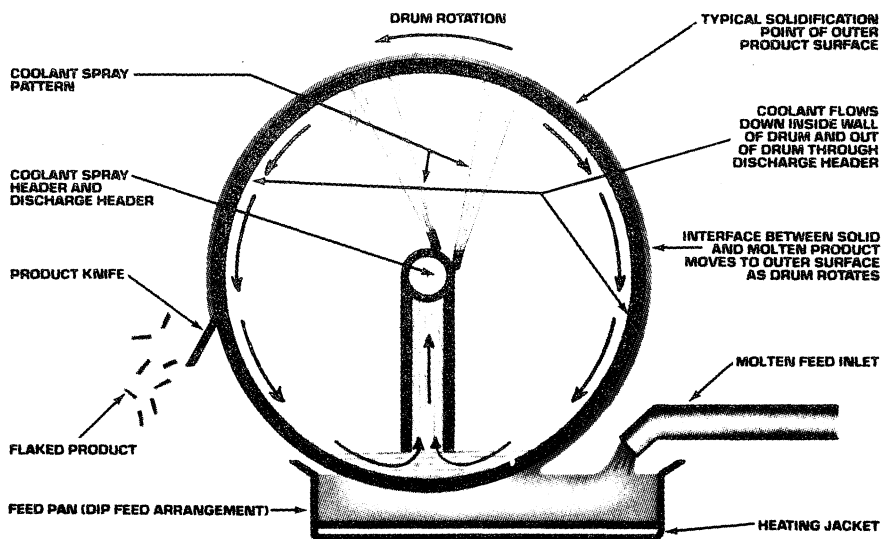


Fig. 6.5. A typical drum flaker operation. (Courtesy Envirotech Corporation.)

TABLE 6.8

Some characteristics of drum dryers [12].

Drum size range	
diameter	2—10 ft
length	2—14 ft
Drum speed	1—10 rev./min
Contact time with heated surface	6—15 sec
Heat transfer coefficient	220—360 Btu/ft ² h °F
Drying capacity	1—10 lb dry product/ft ² h

TABLE 6.9

Qualitative relationship between operating variables and product characteristics for a drum flaker. (Courtesy Blaw-Knox Food and Chemical Equipment Inc., Buflovak Division).

Operating variables	Product characteristics affected			
	Capacity	Flake thickness	Flake size	Flake temperature
Increased drum speed	Increase	Decrease	Decrease	Increase
Increased drum immersion	Increase	Increase	Increase	Increase
Increased feed temperature	Decrease	Decrease	Decrease	Increase
Increased cooling temperature	Decrease	Decrease	Decrease	Increase

TABLE 6.10

Product characteristics and capacity data for some materials treated in belt cooling systems. (Courtesy Sandvik Conveyor, Inc.)

Product	Thickness (in.)	Feed temperature (°F)	Discharge temperature (°F)	Capacity (lb/h/ft ²)
Resins				
Phenolic	0.062	275	110	46
Phenolic	0.048—0.051	280	92	56.8
Sulphur	0.25	290	150	55
Tetrachlorobenzene	0.06	320	70	90
Asphalt	0.125	425	125	18.5
Urea	0.093	375	140	39
Ammonium nitrate	0.063	400	160	90
Chlorinated wax	0.063	300	100	62
Sodium acetate	0.125	180	100	37.5
Butyl phenol	0.050—0.068	230	97	46.5
Hot melt adhesive	0.436	330	103	14.4
Wax blend	0.024	270	85	26.4
Epoxy resin	0.040	350	100	40

Typical Feeding Systems

Typical Discharge Systems

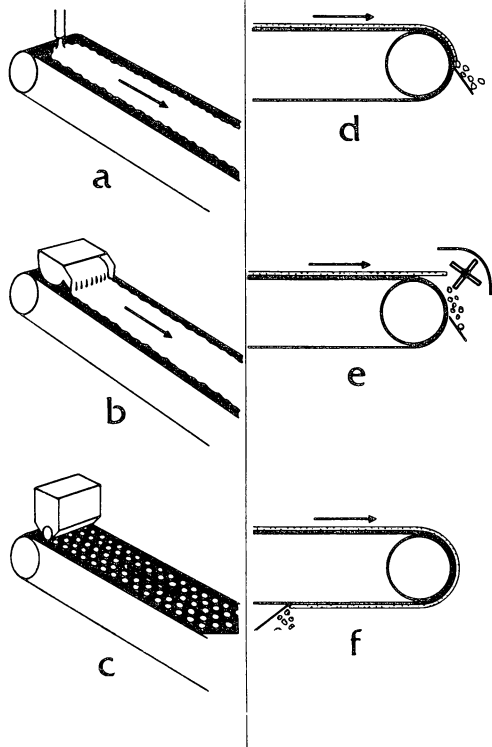


Fig. 6.6. Belt cooling system; typical feeding and discharge arrangements. (a) pipe feed; (b) weir-type feed; (c) drop-forming feed; (d) doctor blade discharge; (e) breaker discharge for size control; (f) discharge from return strand. (Courtesy Sandvik Conveyor, Inc.)

arrangements are possible allowing materials of a wide range of flowability to be treated.

Drum dryers are built as single drum, double drum or twin drum units. In the twin drum unit the tops of the drums rotate away from each other while they rotate toward each other in a double drum unit. Double drum units with top feed at the drum nip are not suited to applications where large hard lumps are possible in the feed or where abrasive solids may settle from the feed. Both conditions may create excessive pressure between the drums.

In *drum flakers*, a thin film of molten feed is applied to the polished external surface of a revolving, internally-cooled drum (Fig. 6.5). Virtually

any molten material that will solidify rapidly with cooling can be treated by this method. Although ambient water is normally the cooling liquid, chilled water and other coolants may be used if lower temperatures are needed. The cooled solid is scraped from the drum as a flaked or granular product.

Unit sizes are similar to those available for drum dryers listed in Table 6.8. The dip feed arrangement of Fig. 6.5 is common but transfer roll, top (nip) feed and other feed mechanisms are also in use. Production capacity and characteristics of the finished product vary from product to product and have a complex relationship (Table 6.9).

3.2. *Endless belt systems*

Molten materials can be cooled to solid products on endless steel belts, as illustrated in Fig. 6.6. Heat is transferred through the belt to a coolant, generally water, which is sprayed on the underside of the belt. A wide choice of belt widths, lengths and speeds lends great versatility to these systems. Some typical products treated in belt cooling systems are listed in Table 6.10, together with product characteristics and capacity data. As with drum flakers, a number of different feeding and product discharge arrangements can be used. Some are shown in Fig. 6.6. Product forms include flakes, pastilles or pellets and slates.

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*Chapter 7***SPRAY AND DISPERSION METHODS**

Granular solids are produced directly from a liquid or semiliquid phase in these methods of size enlargement. Agglomerates are formed in a highly dispersed suspension in apparatus such as spray dryers, prilling towers, spouted and fluidized beds and pneumatic conveying dryers. Feed liquid (solution, gel, paste, emulsion, slurry or melt) is introduced as a spray, dispersed in a gas (generally air) and hence converted to a solid through heat and/or mass transfer. Mechanisms of agglomerate formation include the hardening of feed droplets into solid particles, the layering of solids deposited from the feed onto existing nuclei and the sticking together (coalescence) of small particles into aggregates by deposition of binding solids from the spray.

Other features common to all these spray and dispersion methods include:

- (1) the feed liquid must be pumpable and dispersible;
- (2) the processes are usually amenable to continuous, automated large-scale operation;
- (3) attrition and fines carryover are often a problem and the systems are designed to recover and/or recycle them;
- (4) product size is limited to about 5 mm diameter particles and is often much smaller.

Dispersion methods of simultaneously drying (or hardening) liquids while producing a granular product are applied to a wide variety of materials in the pharmaceutical, food, chemical, mineral and ceramics industries. Rational design of the systems involved requires knowledge of particle size distributions, gas/particle relative velocities and degree of mixing, etc. Seldom is all the required information known and experimental tests are generally required to specify plant for a given application.

1. Spray drying

In this operation, the material to be dried is dispersed into droplet form in a cylindrical (usually vertical) chamber where it is contacted with a large volume of hot gas to evaporate the liquid carrier and produce a dry product. Depending upon feed system and operating variables, a wide particle size range of rounded products can be formed (see Fig. 7.1). Particles as large as 0.5 to 1 mm diameter are possible. When such coarse materials are made spray drying can be considered both a drying and a size enlargement process.

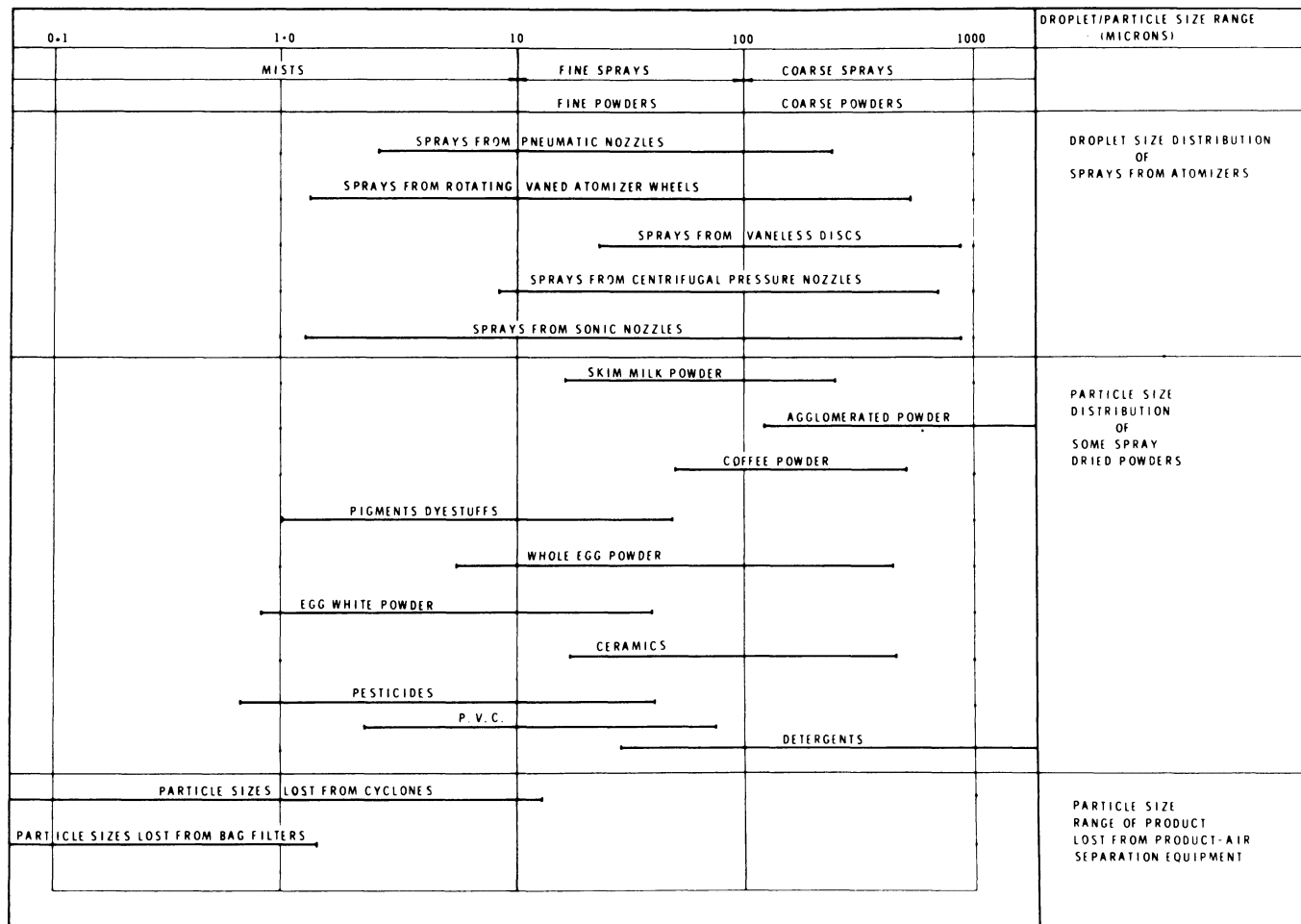


Fig. 7.1. Droplet and particle sizes obtained in spray drying operations [1].

Examples include the manufacture of rapidly dispersible forms of concentrated food products (clusters, agglomerates) and of press feed for various ceramics applications.

1.1. Spray dryer designs [1]

Spray drying involves four fundamental unit processes (see Fig. 7.2):

- (1) dispersion of liquid feed into droplets,
- (2) spray-gas mixing,
- (3) drying of spray droplets,
- (4) separation of dried particles from gas.

Atomization of the liquid feed and contacting the spray with air are the critical features of spray dryers. Dispersion of the feed into droplets is accomplished with either rotary devices or with nozzles. In rotary atomization (Fig. 7.3a), feed is introduced centrally to a wheel (with vanes or bushings) or a disc (vaneless plates, cups, inverted bowls) and is flung off at the periphery where it disintegrates into droplets. Nozzles used can be either single-fluid pressure (Fig. 7.3b) or two-fluid pneumatic (Fig. 7.3c). Thus atomization of the feed can use centrifugal, pressure or kinetic energy. Spray dryers can also be classified according to the product-air flow system used. As depicted in Fig. 7.4, product-air flow can be co-current, counter-current or mixed.

The chamber shape is chosen to accommodate the type of atomizer used. Droplets are ejected in a narrow cone from a nozzle which therefore requires a tall tower to prevent particle contact with the walls before they are dry.

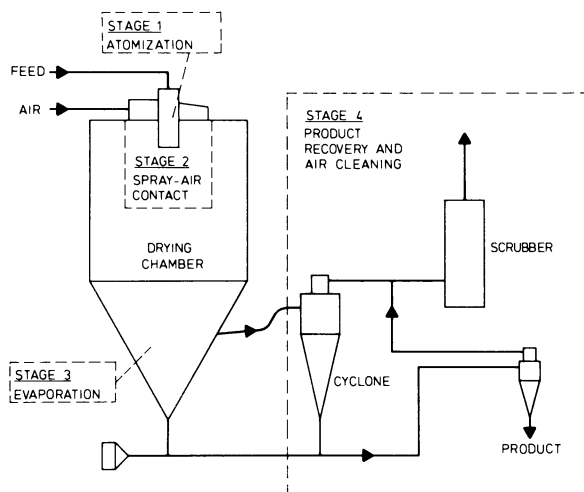


Fig. 7.2. The unit processes of spray drying [1].

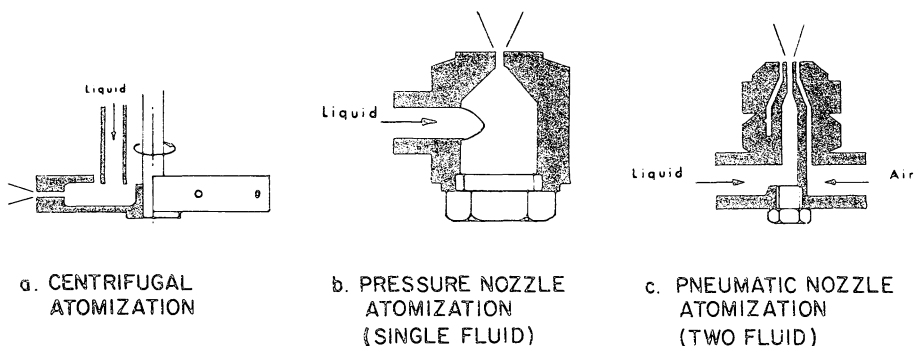


Fig. 7.3. Feed atomization in spray drying. (Courtesy Anhydro, Inc.)

Similarly, a chamber of relatively large diameter but low height is used to conform to the droplet pattern spun horizontally from a centrifugal atomizer.

The water evaporative capacities of the range of standard industrial spray dryers offered by one manufacturer is given in Fig. 7.5 as a function of inlet drying air temperature. Units with capacities up to 40,000 lb/h (18,100 kg/h) or more of water evaporation are available. "Dryer size" in Fig. 7.5 is related to the mass flow rate of drying air; several drying chamber physical "sizes" are possible for each of these air rates, depending on inlet temperature,

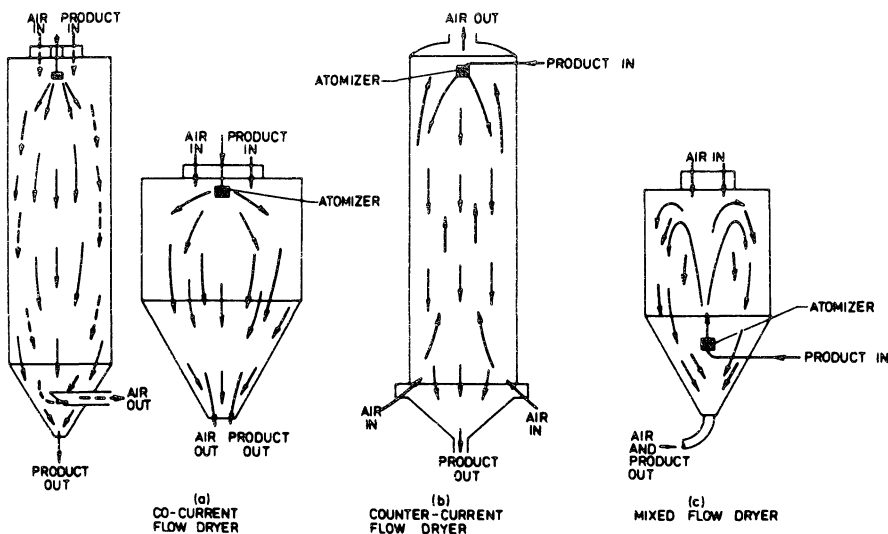


Fig. 7.4. Product and air flow arrangements in spray dryers [1].

Water Evaporation

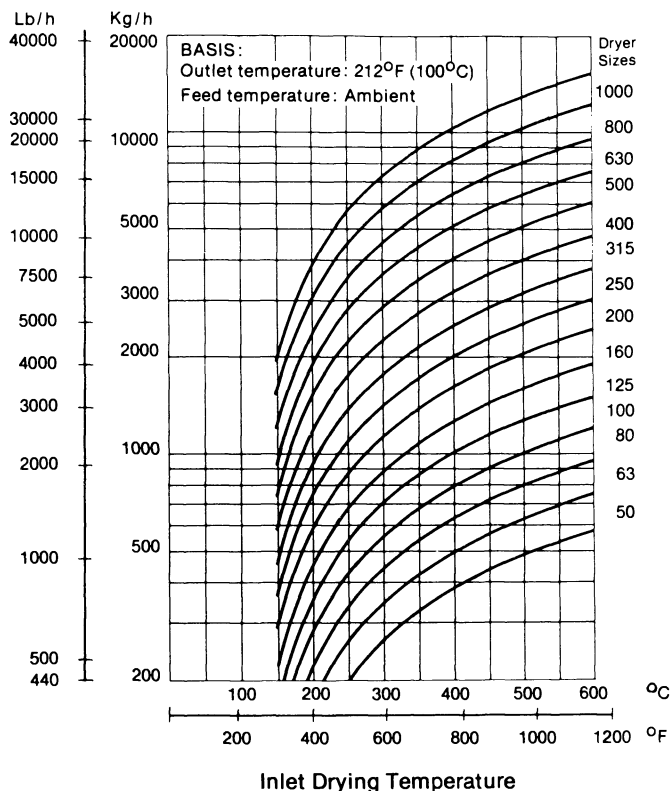


Fig. 7.5. Water evaporative capacities of standard spray dryers fitted with ceiling air dispensers. (Courtesy Niro Atomizer, Inc.)

atomization and other operating conditions. The reader is referred elsewhere [1] for details of theoretical and empirical spray dryer design.

1.2. Spray drying and size enlargement

Spray dried products, in general, are expected to meet a number of requirements. Properties of interest include size and size distributions, bulk density, moisture content, friability, appearance, reactivity and, with food products, aroma and flavour. Control of these properties requires close attention to all four unit processes described in Section 1.1.

From the point of view of size enlargement, the property of most interest is, of course, particle size and its distribution. Some of the variables which can be used to increase the size of spray dried products can be summarized:

- (1) decreased intensity of atomization,
- (2) increased feed viscosity (increased feed solids content, reduced feed temperature, etc.),
- (3) increased feed rate,
- (4) decreased velocity of spray-air contact.
- (5) decreased exit temperature from dryer (higher product moisture, hence tackiness and agglomeration),
- (6) increased tendency to agglomerate (e.g. through presence of natural or added binders).

In addition, the selection and design of the atomizer system is of great importance to product properties. In fact, all the variables noted above interact so that changes in product diameter can be expected to be accompanied by changes in other properties as well. Small-scale experimental tests must generally precede final design.

One example in which a relatively coarse ($250\ \mu\text{m}$ or larger average size) product is produced by spray drying is in the preparation of ceramic press-bodies [2]. A fountain atomizer projects the ceramic slip feed upwards in the drying chamber where it meets incoming drying air and falls back to the bottom of the chamber. (This is the mixed product-air flow arrangement of Fig. 7.4.) This fountain-like spray provides longer residence times and allows coarse powders of the non-heat-sensitive ceramic materials to be produced in small drying chambers. Such installations generally are limited to water evaporative capacities of $2000\ \text{kg/h}$ ($4400\ \text{lb/h}$).

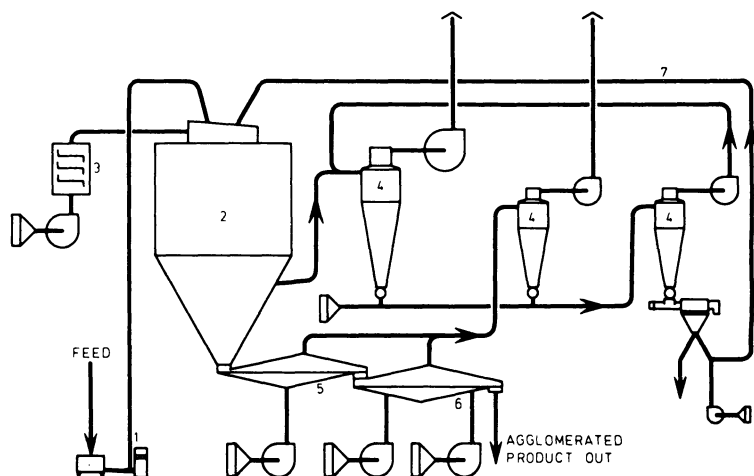


Fig. 7.6. Spray dryer for production of coarse food powders with "instant" properties [29]. (1) Liquid feed system. (2) Spray dryer chamber. (3) Drying air heater. (4) Cyclones for fines recovery. (5) Vibrofluidizer as after-dryer. (6) Vibrofluidizer as after-cooler. (7) Fines return to drying chamber.

Coarse spray dried food powders with “instant” properties (see Table 4.6) are produced directly in the system shown in Fig. 7.6. Instant skim milk powders are manufactured in this system which involves two stages of agglomeration. Initial agglomeration occurs in the atomization zone of the spray dryer where relatively cool air passed around the atomizer retards the evaporation rate of the spray liquid and increases the tendency for fines to agglomerate. Further agglomeration is achieved by operating the spray dryer so that the powder is still moist on leaving the drying chamber. The combination of this moisture level and temperature are controlled (see Fig. 3.3) such that the sticky powder agglomerates to larger sized particles. The agglomerated powder passes out of the bottom of the drying chamber into a first vibrating fluid bed where drying is completed and into a second fluid bed for powder cooling.

When it is not possible to produce a sufficiently coarse or agglomerated product directly by spray drying, agglomerative after-treatments must be used. Methods often used to form larger powder clusters, especially for products such as instantized food powders, detergents, etc., include falling curtain agglomeration (Chapter 4, Section 5), flow mixing agglomeration (Chapter 4, Section 6) and fluid bed agglomeration (Section 3).

2. Prilling

This process is also known as spray cooling, solidification or congealing and shot forming. It is similar to spray drying in that liquid feed is dispersed into droplets at the top of a chamber and the droplets form into a solid granular product during their travel down the chamber. It is distinguished from spray drying by the fact that the liquid droplets are formed from a melt which solidifies primarily by cooling in the chamber with little (if any) drying. Product size is also generally larger (up to about 3 mm diameter) than in spray dried materials. As a result of this relatively large prill size, the process is generally carried out in narrow but very tall towers to ensure that the prills are sufficiently solid when they reach the bottom. Because of the melt feed requirement, prilling is normally limited to materials of low melting point which do not decompose on fusion. Urea and ammonium nitrate fertilizers are traditionally treated by prilling [3,4]. Other prillable materials are listed in Table 7.1.

In most cases air is used as the cooling medium but other gases or liquids may also be used. In the manufacture of lead shot, molten lead is prilled into water. The prilling of calcium nitrate into mineral oil has been described [5]. Prilling into liquid medium requires shorter towers because of the improved cooling by liquids but an extra step to remove the liquid cooling medium is necessary.

TABLE 7.1

Some typical prillable materials. (Courtesy HPD Incorporated.)

Adhesives	Paraffins
Adipic acid	Pentachlorophenol
Alpha naphthol	Petroleum wax
Ammonium nitrate and additives	Phenolic resins, Novalak resin
Asphalt	Pine rosin
Bisphenol-A	Polyethylene resins
Bitumen	Polystyrene resins
Carbon pitch	Polypropylene-maleic anhydride
Caustic soda	Potassium nitrate
Cetyl alcohol	Resins
Coal-derived waxes	Sodium nitrate
Coal tar pitch	Sodium nitrite
Dichloro-benzidine	Sodium sulphate
Fatty acids	Solid glycols
Fatty alcohols	Stearic acid
Epoxy resins	Stearyl alcohol
Hydrocarbon resins	Substituted amides
High-melting inorganic salts	Sulphur
Ink formulations	Urea and additives
Lauric acid	Urea-sulphur mix
Myristic acid	Wax-resin blends
Myristyl alcohol	Substituted aliphatics

2.1. Design considerations [5—7]

A schematic diagram of a prill tower is given in Fig. 7.7. Some details of an installation for fertilizer materials are listed in Table 7.2. Melt is provided to the prilling devices at the top of the tower from reactor/evaporator systems in the case of a fertilizer material such as ammonium nitrate or from a melt tank in the case of fusible materials such as petroleum wax and coal tar pitch. Melt droplets travel counter-current to cooling air and the solid prills are conveyed away from the bottom of the tower to appropriate downstream treatments such as cooling, clay treatment and storage.

As in spray dryers, a variety of devices have been used or suggested for producing droplets from the melt. Centrifugal devices, such as spinning discs and rotating perforated baskets impart an initial radial velocity to the droplets. Such devices require larger tower cross-sections and may lead to inefficient air/droplet contact due to non-uniform prill distribution across the tower [6]. These devices are best-suited to prill tubes of circular cross-section. Atomizing nozzles produce small droplets which are only suitable when fine prills are required.

Vernède [7] states that the two systems most commonly used in the fertilizer industry are nozzles or spray pipes and rotating buckets. For small

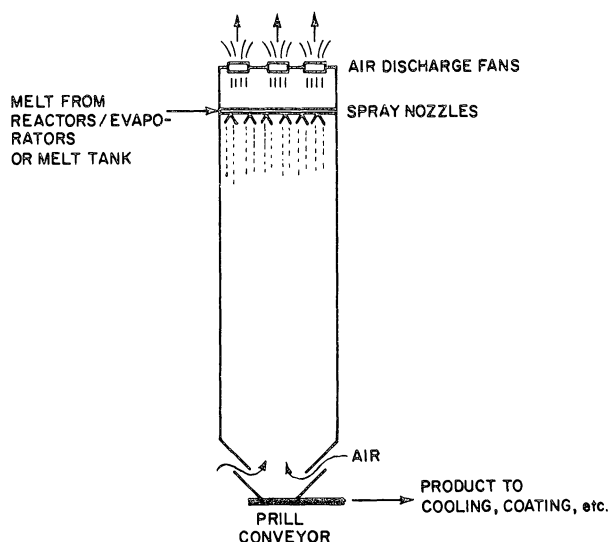


Fig. 7.7. Schematic diagram of a prill tower.

installations, a perforated plate may be satisfactory. Carter and Roberts [6] prefer the slow discharge of the melt through an array of simple orifices. They cite one installation in which the concentrated melt is prilled by gravity through approximately 5000 holes drilled in 5 in. diameter spray roses. Droplets are thus formed by the break-up of the resulting jets producing relatively narrower droplet size distributions and a droplet size which is almost independent of the properties of the melt and the discharge velocity. Droplet

TABLE 7.2

Some characteristics of a typical prilling operation. (Courtesy HDP Incorporated.)

Tower size		
Prill tube height (ft)	130	
Rectangular cross section (ft)	11 by 21.4	
Cooling air		
Rate (lb/h)	360,000	
Inlet temperature	ambient	
Temperature rise ($^{\circ}$ F)	15	
Melt		
Type	Urea	Ammonium nitrate
Rate (lb/h)	35,200 (190 lb H ₂ O)	43,720 (90 lb H ₂ O)
Inlet temperature ($^{\circ}$ F)	275	365
Prills		
Outlet temperature ($^{\circ}$ F)	120	225
Size (mm)	approximately 1 to 3	

size is approximately equal to twice the diameter of the undisturbed jet.

One of the more critical feed properties in the case of ammonium nitrate prilling is the moisture content of the melt. Evaporation of the melt to nearly 100% solids produces hard, non-porous prills. Larger water contents yield porous, less dense prills.

For viscous, high-melting and sticky materials such as resins, a hot chamber method, the Thermaprill System [4], has been developed. Prilling nozzles are mounted atop the prilling tower in a radiant-heat thermal conditioning chamber through which no cooling air is allowed to flow. The arrangement prevents the nozzles from plugging or freezing and provides a zone in which streams from the orifices can break up into droplets before solidification begins. Fewer fines are claimed as is the ability to prill many materials not treated this way in the past.

2.2. Tower size

The two principal tower dimensions to be determined are the cross-sectional area and the free-fall height. Restraints which must be considered in sizing the *cross-sectional area* include:

(1) The production rate and the number of prilling nozzles required to achieve this production.

(2) The minimum area required to allow sufficient air through the tower to satisfy the cooling load at a velocity which is sufficiently low to prevent excessive prill carry-over.

(3) the maximum air temperature allowable at the top of the tower.

The *free-fall height* is determined by the retention time necessary to cool the desired prill size to the required temperature at the existing air velocity. The effect of the counter-current air flow which may be of the order of 3–7 ft/sec (0.9–2.1 m/sec), is considered to be negligible by Vernède [7]. Theoretical calculations are possible to determine tower height with reasonable accuracy. These are simplified in the case of prilling towers compared with spray dryers, since droplet motion under evaporating conditions must be taken into account in the latter case, with the resulting effects on droplet trajectory and heat and mass transfer. Simple parallel streamline flow of both droplets and air is a reasonable assumption in the case of prilling towers compared with the more complex rotational flows produced in spray dryers.

In prilling operations, prill sizes encountered during gravity fall lie in the transition region between laminar and turbulent flow. As a conservative approach, however, viscous forces may be neglected [6] and the velocity of fall calculated [8] from eqn. (1):

$$V = \frac{\alpha e^{2\alpha\beta t} - 1}{\beta e^{2\alpha\beta t} + 1} \quad (1)$$

in which

$$\alpha^2 = g \left(1 - \frac{\rho}{\rho_s} \right) \quad \text{and} \quad \beta^2 = \frac{c_1 \rho s}{8m}$$

where c_1 is the inertial drag coefficient, g is the acceleration due to gravity, m is the mass of a prill, s is the prill surface area, t is time of travel from rest, V is the prill velocity relative to the air, ρ is the air density, and ρ_s is the prill density.

Assuming spherical prills and heat transfer by forced convection, the heat transfer coefficient between the prill and air can be determined from a relation of the form:

$$Nu = F(Pr, Re) \quad (2)$$

where Nu , Pr and Re are the Nusselt, Prandtl and Reynolds numbers, respectively.

Details of the functional relationships in eqn. (2) are available in the literature (see, for example, references 9 and 10).

With the known physical data for a given system (specific heats, heats of crystallization and transition, etc.), eqn. (1) and (2) can be used to calculate prill temperature, velocity, distance travelled, etc., in a stepwise iterative procedure down the height of the tower. Computer calculation is indicated [6]. The optimization of tower height for ammonium nitrate prilling (with consideration of the balance between complete product cooling in the tower and partial tower cooling with completion in an external cooler) has been described [6] using such a calculation routine. Prill size and tower throughput were both found to influence the economic optimum tower height. Operating ammonium nitrate prilling towers ranging over the wide height range of 22 m to 72 m free-fall were cited.

Other papers have considered the theoretical calculation of prill trajectories [11] and the design of vessels for prilling into liquid cooling medium [5].

3. Fluid bed spray granulation

This operation, as in spray drying and prilling, converts pumpable and atomizable feed liquids (solution, slurry, paste, melt) to granular solids in one step through drying. Atomized feed droplets impinge onto a fluidized bed of hot seed particles and solids are deposited by drying together with chemical reaction in some cases. The seed bed particles grow either by coalescence of two or more particles held together by a deposited binder material, or by layering of solids onto the surface of individual particles. As in agitation methods of size enlargement (Chapter 3, Section 1.1), the coalescence mechanism yields more irregular or cluster-type agglomerates.

Because of their ability to deposit multiple layers of solids on a given par-

ticle, fluidized systems can produce larger granules than spray dryers. The product is thus less dusty and the longer residence times possible mean that larger dryer loads with less concentrated feed liquors can be handled. Since the drying particles are less dispersed in fluid beds, smaller equipment is needed [12].

Fluidization techniques have been known and used for three or four decades [13,14]. Although particle formation and growth during fluidized bed operation has been known since the earliest applications of the technique, the particle forming aspect has been somewhat secondary to other process objectives such as chemical reaction. This is true, for example, in the fluid coking process [15] and in fluid-bed incineration [16] and calcination [17]. As a size enlargement operation, the application to produce tablet granulations [18–21] was one of the earliest; but more recent applications, such as the preparation of press feed for ceramic tiles, indicate that this aspect of fluid bed technology will become increasingly important [22].

3.1. Process description [22]

The various components of a typical fluid bed spray granulation unit are depicted in Fig. 7.8. Fluidizing gas (usually air) at 2 or 3 psig (14 to 21 kPa) is heated externally to the fluid bed and passes to the base of the unit. Here a suitable distributor such as a perforated plate or tubes with nozzles passes the gas to the particle bed uniformly over its cross-section. Jets formed at

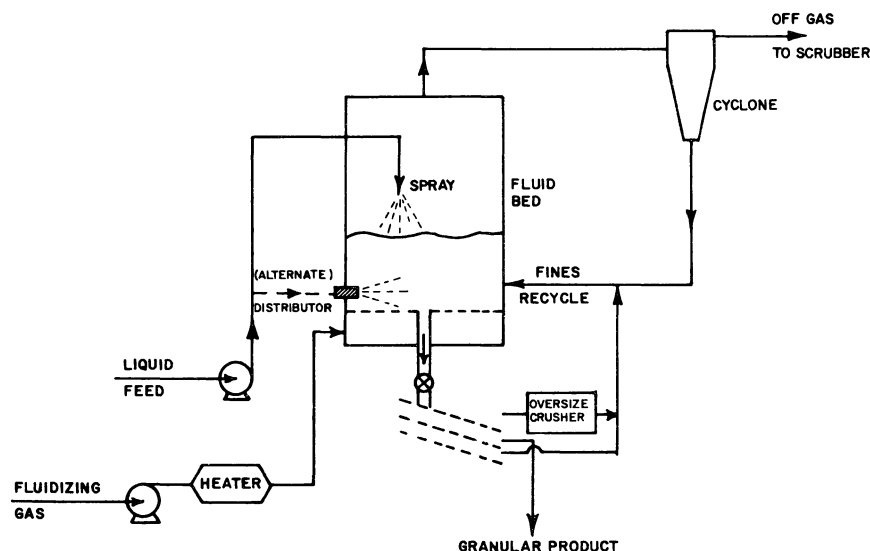


Fig. 7.8. Fluid bed spray granulation.

the distributor may be directed to adjacent deflector plates to break up large particles and lumps which can accumulate at the bottom of the bed.

Liquid feed is most often dispersed into the bed by a two-fluid spray nozzle which is flexible in operation and less prone to blockage, especially if heated air is used to atomize crystallizing liquors. Liquid feed may be directed onto the bed surface from the space above the upper bed surface or introduced directly beneath the surface of the bed material. The liquid spray is less likely to dry as a spray before contacting the bed particles in this latter arrangement.

The fluidizing chamber can consist of more than one compartment. This provides different process conditions (e.g. temperature, moisture level, gas velocity, etc.) as material flows through the bed and encourages conditions closer to plug flow for the granular solids, leading to a more uniform product size distribution [23]. Fluidizing gas velocities sufficiently high to give vigorous particle movement are necessary to prevent lump formation.

Product discharge is accomplished through an opening below the bed surface. The discharge location can be selected to remove preferentially larger granules which accumulate deeper in the bed. Withdrawn product is often screened, with crushed oversize being recycled to the bed together with the undersize. Exhaust gases leaving the bed surface may pass through an enlarged particle disengaging section. Elutriated particles can also be collected for recycling by external bag filters or cyclones. The hot gases may also pass through a wet scrubber fed by incoming feed liquor to improve heat economy while preconcentrating the liquid.

3.2. System and operating variables; factors affecting product size

Many of the principles already described in Chapter 3 concerning the effect of process parameters and the control of tumbling agglomerators also apply to fluid bed granulation. For example, the control of granule nucleation is essential to stable operation. In continuous operation such as that in Fig. 7.8, the rate of production of stable new seeds must equal the rate of production of product size granules. New seeds are generated by a number of mechanisms, including drying of liquid feed to solid before contacting the bed, by attrition and fracture of bed particles, by recycle of crushed oversize product and by introducing new solid particles as part of the feed. Also similar to other agitation methods of size enlargement, increased intensity of agitation by increasing the fluidizing gas velocity leads to smaller particle size in the bed.

The effects of a number of parameters on the properties of fluid bed granulation products [19,22] are summarized below. These generalizations require experimental testing, however, for each spray granulation application.

3.2.1. Liquid feed

Increase in the rate of liquid feed addition and in its solids or binder content generally produces larger, stronger and more dense granules. There is, of course, an upper limit to the liquid feed rate set by increasing cohesion of bed particles and a tendency for the bed to slump or defluidize.

In some cases, as the solids content of the feed increases, the feed may tend to spray dry in the space above the bed, forming new seed particles and smaller particle size in the bed.

3.2.2. Spray characteristics

Coarser spray droplets yield larger granules which tend to a “raspberry” coalesced, rather than an “onion skin” layered, structure. This effect is diminished the larger the ratio of granule size to droplet size.

With two-fluid sprays, the atomizing air tends to dry the liquid feed. Hence an increase in air rate or its temperature reduces the ability of the spray to wet and penetrate the bed material, leading to smaller bed particle size.

Larger particles are produced as the wetted fraction of the bed material is decreased. Wetting and penetration of the sprayed portion of the solids is enhanced in this way. Thus granule size increases as the number of spray nozzles is decreased, a narrower spray angle is used or the spray is positioned closer to the bed surface.

3.2.3. Fluid bed conditions

As noted already, the bed particle size changes inversely with the intensity of fluidization. Hence reduced gas velocity increases granule size and coalesced structures are favoured, while layer growth and smaller particles result at high velocities. A lower limit exists for the gas velocity below which particle movement is inadequate and defluidization occurs.

Generally bed temperature and granule size are inversely related where bonding occurs by solids deposited through drying. If melts are formed at the higher temperature, however, or if a melt is sprayed as liquid feed and bonding occurs through cooling, higher temperatures may yield larger particles.

3.2.4. Outlet gas conditions and recycle of fines

When the spraying system is located above the bed surface, considerable drying of the spray may occur before it contacts the bed particles. The extent of this drying depends, among other factors, upon the difference between the outlet gas temperature and its dew point. The drying potential

increases with this difference and more new particles (seeds) are formed from the spray, leading to finer particle size in the bed.

Similarly, the degree of grinding of oversize product and its recycle back to the bed together with fines strongly influences the creation of new granule centres. Control of the fineness and amount of recycled solids is the most satisfactory way to control particle size and stabilize granulator operation [12,22].

3.3. Design and performance information

Preliminary design of fluid bed granulators can be done on the same basis as for fluid bed dryers in general. Material and thermal balances allow the gas flow to be determined and correlations are available [13,14] to yield the fluidization velocity for selected bed voidage. The distributor area can then be calculated to give this velocity for the required gas flow. Knowledge of the kinetics of the drying process is necessary to calculate the required bed volume from which the dryer height can be determined with a suitable "free-board" allowance. Calculation procedures and examples are available elsewhere [20,21,24,25].

Detailed design of fluid spray dryers must, however, be accompanied by experimental work. It has been noted [22] that even quite similar materials may behave very differently in a spray granulator. Experimental work will indicate such information as the "natural" mechanism of particle growth (coalescence vs layering), attainable product sizes, the preferred temperature and moisture conditions for the bed material, suitable ranges of bed voidage

TABLE 7.3

Characteristics of batch fluid bed spray granulators to produce tablet granulations in the pharmaceutical industry. Flowsheet given in Fig. 7.9.

	Approximate range
Batch load, dry basis (lb)	20—400 *
Volume of container for static bed (ft ³)	2—15
Fluidizing air fan (hp)	5—25
Air (steam) heating capacity (Btu/h)	70,000—600,000
Drying air temperature (°C)	40—80
Granulating liquid spray **	Two fluid nozzle
air volume	0.5—2 SCFM
liquid volume	500—1500 cm ³ /min
Batch processing time (min)	30—50
Average granule size	24—8 mesh

* Batch capacity exceeds 1500 lb in largest modern units.

** Typical granulating liquids are gelatin or sodium carboxymethyl cellulose solutions.

TABLE 7.4

Granular products from fluidized bed incineration [16].

Type of sludge	Incinerator size	Bed temperature	Capacity	Granular product composition
Oil refinery waste sludge (85—95% water)	40 ft high; 20 ft I.D. at base increasing to 28 ft at top	1330° F	31×10^3 lb/h of sludge	Start-up material was silica sand; replaced by nodules of various ash components such as CaSO_4 , Na, Ca, Mg silicates, Al_2O_3 after operation of incinerator
Paper mill * waste liquor (40% solids)	20 ft I.D. at top	1350° F	31×10^3 lb/h	Sulfur added to produce 90—95% Na_2SO_4 and some Na_2CO_3

* Flow sheet, Fig. 7.10.

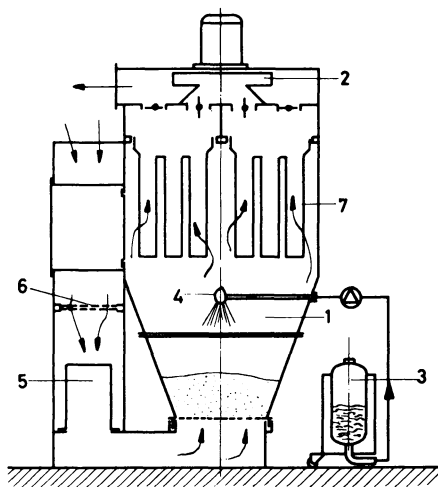


Fig. 7.9. Batch fluid bed spray granulator used to produce tablet granulations in the pharmaceutical industry. Air-flow necessary for fluidization is generated by a suction-fan (2) mounted in the top portion of the unit, directly driven by an electric motor. The air being used is heated to the desired temperature by an air heater (5). Prefilters remove all impurities at the air inlet (6). The material to be processed has been loaded into the material container (1). The container bottom consists of a perforated plate above which a fine mesh stainless steel retaining screen is fitted. Exhaust filters (7) mounted above the product container retain fines and dust. The granulating liquid (3) is sprayed as a fine mist through a mechanical or pneumatically actuated nozzle (4) onto the finely dispersed, fluidized material to form the desired agglomerates. (Courtesy Aeromatic AG.)

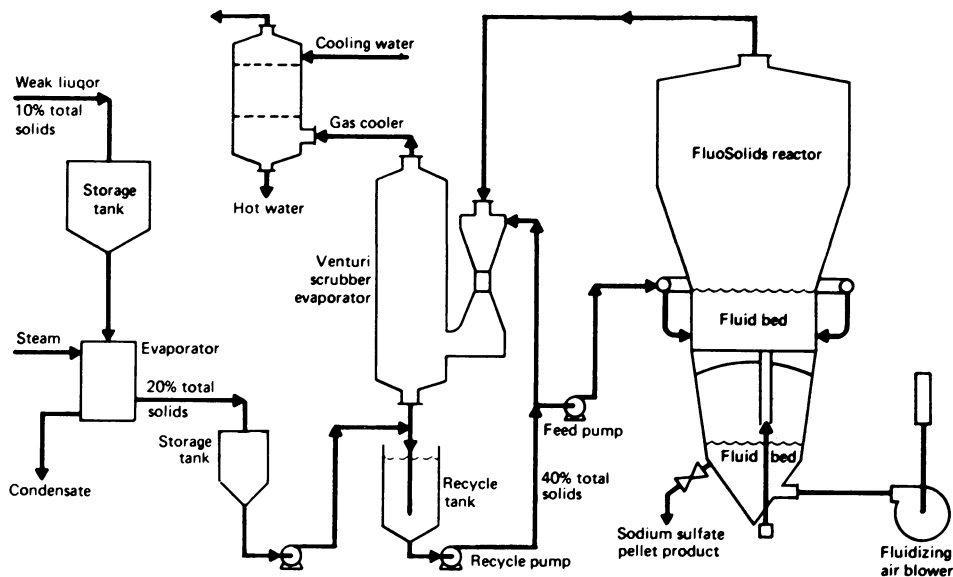


Fig. 7.10. Fluid bed incinerator flowsheet for paper mill waste liquor [16].

or density and estimates of drying rate. For continuous operation, the stability of the operation (especially the need to add seed fines and/or pulverized recycle, and at what level) can only be determined by experiment.

Performance data for two industrial versions of the fluid bed spray granulation technique are given in Tables 7.3 and 7.4. Corresponding equipment diagrams are found in Fig. 7.9 and 7.10, respectively. In Table 7.3 data are listed for a range of batch spray granulators available for the production of tablet granulations in the pharmaceutical industry. In this application, the fluidized bed granulator combines into one step several of the individual operations (e.g. size control, drying, blending) normally used in other granulation techniques. Table 7.4 contains data on the fluid bed incineration process. Although the main objective of this process is disposal of waste sludges, the granular ash product may often be a salable chemical by-product. In this secondary aspect fluid bed incineration can be considered as a size enlargement process.

The continuous clay spray granulation process to achieve a clay particle size distribution and moisture content suitable for pressing into two-coloured ceramic tiles is carried out in a system similar to that in Fig. 7.8 [22]. The fluid bed in this case, however, is divided into two compartments with small agglomerate nuclei being formed in the first stage. These are then coalesced into “raspberry-type” structures about 1 mm diameter in the second stage to yield a product suited to pressing. To achieve this result, the following condi-

tions are used:

Moisture content of clay

first stage	3 to 5%
second stage	6 to 9%
Air inlet temperature	360°C
Temperature at air distribution plate	210°C
Recycle to product ratio (exclusive of cyclone fines)	2 : 1 to 3 : 1

In addition, the clay slip feed nozzles are kept very close to the bed surface (approx. 10 cm or 4 in.) in the second stage to encourage coalescence of the seed particles formed in the first compartment. Maximum production in this type of spray granulator is now of the order of 50,000 kg/h (55 tons/h) of product.

4. Spouted bed granulation

This process is similar to fluid bed granulation in that pumpable and atomizable feed liquids are converted to granular solids in one step by spraying feed liquid onto hot seed particles where feed solids are deposited by drying. The operations differ in the method for agitating the growing particles in the bed. As shown in Fig. 7.11 hot spouting gas is injected as a single jet into the

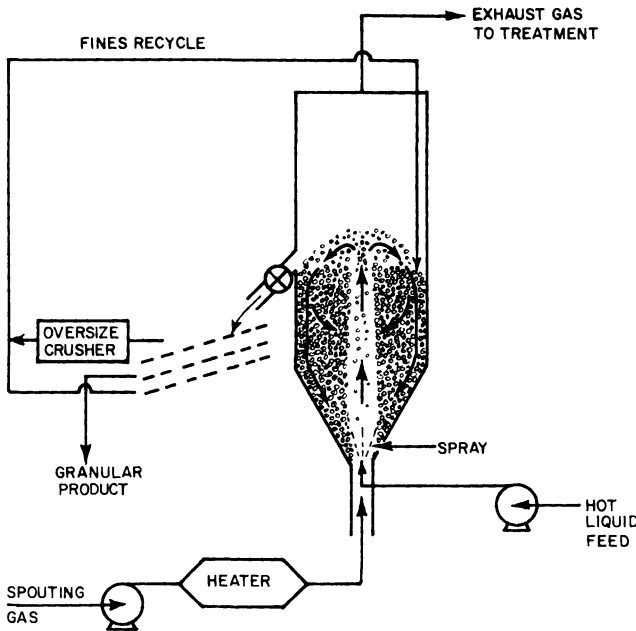


Fig. 7.11. Spouted bed granulation.

TABLE 7.5

Spouted bed granulation data for some agricultural chemicals [26]. Performance data reported by Berquin [27].

Material	Feed solution		Product		Air temper- ature (°C)		Air flow rate (m ³ /sec)	Capacity (Mg/h of product)	Weight of bed of seed granules (Mg)
	Moisture (%)	Temperature (°C)	Size (mm)	Moisture (%)	Inlet	Outlet			
Complex fertilizer (nitro-phosphorus)	27	cold	3—3.5 (90%)	2.4	170	70	13.9	4	—
Potassium chloride	68	cold	4—5 (oversize <5%)	—	200	60—65	13.9	1	1
Ammonium nitrate	4	175	2.5—4	0.2	cold	55	13.9	9.5	1.5
Sulfur	0	135	2—5	0	cold	—	0.011 *	0.04	0.008

* Injecting 1 liter/h water as spray into the spouting air reduced the air requirement to 0.007 m³/sec for the same product output.

conical base of the granulation chamber causing the bed material to circulate much like a water fountain. Particles are carried up the central spout as a dilute phase until they lose their momentum and fall back onto the top of the bed around the outer periphery. They recirculate back down the column as a dense moving bed and are directed back into the gas stream by the conical base of the apparatus. Liquid feed, injected as a spray into the base together with the hot spouting gas, deposits a thin layer of liquid onto the recirculating seeds. Feed solids deposit by drying from the liquid onto the particles as they cycle up the spout and down the annulus.

Spouted beds were originally developed as an alternative to fluidized beds as a means of contacting gas with solids. The operation of fluidized beds becomes less effective when large particles (greater than, say, 1 mm diameter) are to be treated. The gas-solids contacting efficiency is impaired at larger particle sizes as more and more gas bypasses in the form of large bubbles. In contrast, about 1 mm diameter particles is the *minimum* size for which spouting appears to be practical [26]. Thus, spouted bed granulators allow larger granules to be formed than is the case for fluid bed granulators. Other advantages of spouted bed compared with fluid bed granulators have been discussed in the recent book by Mathur and Epstein [26]. These include:

(1) Higher permissible inlet gas temperatures due to rapid evaporation of the spray liquid in the high gas velocity region at the base of the spout with consequent rapid cooling of the inlet gas.

(2) Formation of well rounded and uniform granules through the cyclic movement of the solids which favours growth by layering as opposed to coalescence of bed particles.

(3) Narrow product size distribution, in part due to a classification effect at the top of the annulus which allows the outlet pipe to be located so as to remove primarily the largest granules.

(4) Avoidance of problems with distributor plate scaling, especially in cases where granulation is combined with both chemical reaction and drying.

The comments made in Section 3 on the design of fluid bed granulators apply equally to spouted bed systems. Information specific to the theoretical design and analysis of spouted beds is available in ref. [26]. Performance data for the spouted bed granulation of some agricultural chemicals are given in Table 7.5.

5. Pneumatic conveying or flash drying

In this technique, wet solids are dried almost instantaneously by dispersing and conveying directly in a high velocity stream of hot gas. Although the major objective is to dry the wet material, granular products in the range 0.1 to 1 mm diameter are produced and in certain applications where cohesive fine wet materials are treated, the method may offer an alternative to more

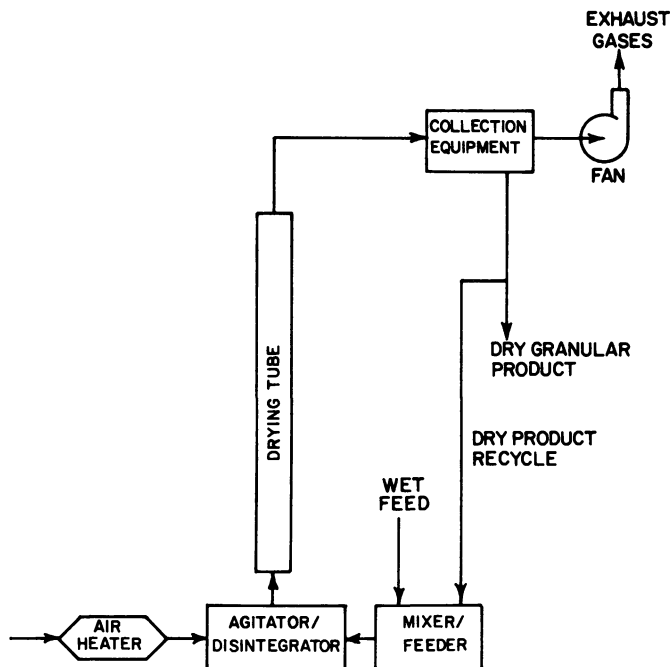


Fig. 7.12. Principal components of a flash dryer with dry product recycle for sludges, cakes and pastes.

common size enlargement methods. For example, filter and centrifuge cakes, thick slurries, sludges, pastes and similar materials can be reduced to a dry granular product in the flash dryer represented schematically in Fig. 7.12. Cohesive materials are handled by mixing them with recirculated dried product, for example in a paddle mixer, followed by disintegration in a mill to disperse them in the hot gases. In this way a maximum of wet surface is exposed to complete the drying in the short residence times (1 to 10 sec) available.

Because of the high evaporation rates achieved, high inlet gas temperatures up to about 1400°F (760°C) can be employed even with flammable materials. High thermal efficiency is achieved. The drying tube is generally vertical although a number of dryer configurations are used (see, for example, ref. 28). Evaporative capacities up to 20,000 lb/h (9100 kg/h) of water, or more, are available in flash drying units. Cohesive materials handled in this way include clays, diatomaceous earth, starches, and byproduct and waste materials such as sewage sludge.

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AGGLOMERATION IN LIQUID MEDIA

1. Introduction

Although fine *dry* powders present difficulties such as dusting losses and other handling hazards, finely divided materials *in liquids* are also difficult to deal with. The size of the individual particles is often so small that methods to capture them (such as filtration or gravity sedimentation) are not feasible unless some form of size enlargement is applied.

Size enlargement of fine particles in liquid suspension can be accomplished in a number of ways. Electrolytes can be added to a suspension to cause a reduction in zeta potential and allow colliding particles to cohere. Examples include the use of trivalent aluminum and iron ions to flocculate the particles responsible for the turbidity of many water supplies and the flocculation of metallurgical slimes by pH adjustment to the isoelectric point. Alternatively, polymeric flocculants can be added to suspensions to bridge between the particles. A wide range of such polymeric agents [1] is available today to aid the removal of fine particles from water.

In conventional flocculation procedures, interparticle bonding forces are relatively small and result in rather weak, voluminous, cluster-type agglomerates. The objective of the procedure is simply to remove the fine particles from the liquid medium. The present chapter is not concerned with these traditional methods to remove solid from liquids but deals with those procedures in which stronger interparticle bonding and specialized equipment are used to form larger and more permanent agglomerates in liquid suspensions. The objectives of these latter methods are somewhat broader than those of the former and include the production of granular (including spherical) material, displacement of as much suspending liquid as possible from the product and the selective agglomeration of one or more components of a multiparticle mixture, in addition to separation of the particles from the suspension. Information on the more conventional flocculation procedures can be found elsewhere in the literature [1,2].

2. Agglomeration by immiscible liquid wetting

Fine particles in liquid suspension can readily be formed into large dense agglomerates of considerable integrity by the addition of suitable amounts of

a second or bridging liquid under appropriate agitation conditions. This second liquid must be immiscible with the suspending liquid and must wet preferentially the solid particles which are to be agglomerated.

A simple example of immiscible liquid wetting is the addition of oil to a fine coal suspension to agglomerate and remove the carbon constituents while the inorganic impurities (ash constituents) remain in suspension and are rejected. A number of such coal cleaning processes, such as the Trent Process [3], the Conventol Process [4] and the Spherical Agglomeration Process [5], have been developed and used in this century. As discussed below, developments of the latter process have shown that many other applications are possible for immiscible liquid wetting.

2.1. General characteristics [6]

The phenomena which occur as progressively larger amounts of bridging liquid are added to a solids suspension are represented in Fig. 8.1. At low levels of bridging liquid, only pendular bridges (see Section 2.4, Chapter 2) can form between the particles with the result that an unconsolidated floc structure exists. As seen in the lower part of Fig. 8.1, a loose settled mass of volume larger than that of the unflocculated particles, but of faster settling rate, results. As the funicular region of bridging liquid levels is reached, the flocs consolidate somewhat and lower settled volumes are recorded. Some compacted agglomerates appear and increase in number until about midway in the funicular region the whole system has been formed into "microagglomerates". As the amount of bridging liquid is increased, the agglomerates grow in size and reach a peak of strength and sphericity near the capillary region.

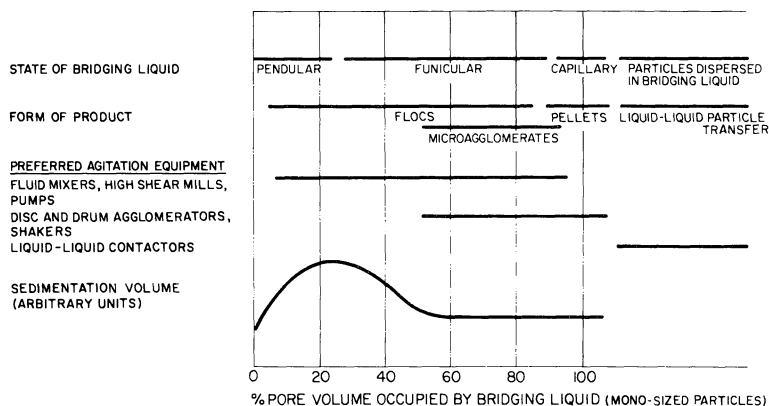


Fig. 8.1. Agglomeration "phase diagram" [6]. Phenomena which occur as increasing amounts of an immiscible wetting liquid are added to a solids suspension. A typical example is fine coal in water to which an organic liquid is added as bridging agent.

Beyond this region the agglomerates exist as pasty lumps; the solids are then essentially dispersed in the bridging liquid.

It is evident that a variety of agglomerated products (ranging from flocs through larger and denser agglomerates to a fluid mass with particles suspended in bridging liquid) can be formed from particles in liquid suspension by wetting with a second immiscible liquid. Each type of agglomerate has applications particularly suited to it as summarized below.

2.1.1. Removal of fine particles from liquids

This application of immiscible liquid wetting is that which has seen the widest industrial usage. Particles in liquid suspension, down to the sub-micron range if necessary, can be agglomerated to larger entities and rapidly and cleanly removed from the suspension. Table 8.1 summarizes a number of such applications in which various means, such as sedimentation, tabling, screening and cyclone separators were used to recover the size-enlarged particles. As larger and denser agglomerates are formed at higher bridging liquid levels (in accordance with Fig. 8.1), the amount of suspending liquid retained by the agglomerates is reduced. This is due not only to the displacement of suspending liquid from agglomerate interiors by the adsorbed bridging liquid, but also to the reduced surface area of the products and consequently reduced amounts of surface-held suspending liquid.

2.1.2. Pelletization and sphere formation

Many of the agitation methods of size enlargement described in Chapters 3 and 4 can be carried out under a blanket of suspending liquid immiscible with the bridging liquid being used. Such pelletization from liquid suspension has certain advantages, such as reduction of dust nuisance which may be a problem with conventional "dry" methods and the ability to agglomerate directly from liquid suspension where the feed material is already in this form. In addition, the liquid blanket may be helpful in dispersing the bridging liquid uniformly and in developing improved agitation conditions for agglomeration since the suspended solids follow the fluid elements during mixing. Highly spherical agglomerates can be formed in this way using intensive mechanical agitation. Further details of these methods of pelletization and sphere formation are available in the literature [7–9].

2.1.3. Selective agglomeration

This is the feature which distinguishes immiscible liquid wetting from other size enlargement techniques. One or more components of a complex solids mixture can be selectively agglomerated and removed from suspension while other components remain. Where the natural wetting properties of a particulate component do not allow its separation from a suspending liquid and/or from other particles of a mixture, surface conditioning agents may be used to modify its surface properties and allow the desired separation. Table

TABLE 8.1

Some examples of the removal of fine particles from liquids by immiscible liquid wetting [6]

Suspension treated	Bridging liquid	Equipment
Calcium sulphate in phosphoric acid (wet process for phosphoric acid)	Petroleum sulphonate surfactants/hydrocarbon liquid	Conditioned in mixer, pellets formed by tumbling
Iron ore in water (tailings slurry)	Oleic acid/motor oil	Mixing tank
Fine coals in wash plant slurries	Light hydrocarbon oil for selective flocculation; heavy oil for balling.	High intensity mixer for flocculation; beneficiation, balling on a disc.
Soot in water	Heavy oil	Rotating annular mixer/pelletizer.
Phosphates ores in water	Fatty acid/fuel oil	Conditioned in mixer, separated on tables
Oil dispersed in water	Finely-divided hydrophobic solid (eg coal) acts as emulsion breaker.	Turbine mixer
Peat moss in petroleum solvent.	Aqueous salt solutions	
Sodium lignate in water, pH = 10.	Rubber latex, acidify to pH = 3.	Turbine mixer
Silica aquagel, preparation of dry colloidal silica	Ammonium oleate/hydrocarbons	Turbine mixer

8.2 summarizes a number of examples of ores, fossil fuels, and other particle mixtures in which selective agglomeration was used to effect separations.

Selective agglomeration can be carried out across the whole range of bridging liquid levels represented in Fig. 8.1. In the pendular and funicular regions, selective flocculation occurs usually with the aid of vigorous agitation. Beyond the capillary region, a collecting liquid may be contacted with the particle suspension using a gentle kneading-type agitation with the selected particles being transferred from the original suspension into the agglomerating liquid.

2.2. Processes and equipment [5,10]

It will be evident from the previous section that many potential uses exist for the recovery of fines from liquid through preferential wetting by a second liquid. The agglomeration of carbonaceous fines (especially coal parti-

Reference	Remarks
21	Ashed CaSO_4 pellets contained 1.1 to 1.8% P; greater than 99% recovery of phosphoric acid from a semi-gelatinous precipitate
22,23	Agglomerates removed by cyclone separator which may also aid in forming agglomerates.
5,24,25	Waste fine coals upgraded to acceptable ash levels and desirable pelletized form.
12	Wash-water stream from oil gasification process. Soot removal down to less than 5 ppm.
26	“Agglomeration Tabling” applied to phosphate potash minerals. Selective flocculation followed by separation on tables. Addition of powder which is agglomerated by dispersed oil and causes emulsion breakdown.
27	Recovery of peat moss following solvent extraction of resins.
28	Lignin reinforcement of rubber.
29	Dewatering of gelatinous precipitate to form high surface area powder.

cles) is an application of great interest today and will be used to illustrate process flowsheet and performance data.

Increasing quantities of fines in water suspension must be processed today during coal preparation. These fines result from natural degradation, increasingly-mechanized mining methods and the finer grinding necessary to liberate impurities from lower quality coals. Coal particles are readily agglomerated and recovered from aqueous suspension upon agitation with many different oils as collecting liquids. Inorganic or ash-forming constituents remain in suspension and are thus rejected.

The sequence of process operations for coal agglomeration is depicted in the generalized flow diagram of Fig. 8.2. Possible equipment for each step is also indicated. In general, equipment well-known in the chemical and mineral industries can be applied in the process. The operations consist of selective flocculation or microagglomeration, agglomerate recovery with simultaneous impurity rejection, further size enlargement of the recovered and

TABLE 8.2

Examples of surface conditioning and selective agglomeration by immiscible liquid wetting [6].

Material treated and objective	Suspending liquid	Conditioning agents and/or other additives	Collecting liquid	Reference
Barite in mill tailings; upgrading	Water	Sodium dodecyl sulphate, sodium silicate, acid wash of agglomerates	Still bottoms	30
Brine (NaCl) solution, removal of colloidal iron oxides	Saturated NaCl solution	Tall oil	Crude oil, sulphonated petroleum oil	31
Coal, bituminous, separation of pyrite and other ash constituents	Water	Iron-oxidizing bacteria, alkali	Petroleum distillates	32
Coal, bituminous, balling of ash constituents	Petroleum distillates	Balling nuclei added	Water	33
Coal, subbituminous and lignitic	Water	Coke oven tar, pitch, petroleum crudes and fractions	Hydrocarbons	5
Germanium in carbonaceous sandstone	Water	Coal tar, Na_2CO_3 and sodium silicate	Petroleum still bottoms	34
Glauber's salt ($\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$), removal of siliceous matter	Saturated Na_2SO_4 solution	Various amines	Hydrocarbon	35
Gold ore for upgrading	Water	Sec-butyl xanthate	Still bottoms	36
Graphite—sulphur mixture	Light petroleum distillate	Tannic acid in collector	5% aqueous tannic acid	37
(1) Microagglomeration and removal of graphite		Aerofloat 15	Dilute NaOH solution	37
(2) Microagglomeration and removal of sulphur				

Graphite -- zinc sulphide -- calcium carbonate mixture	Water			
(1) Microagglomeration and removal of graphite		None	Nitrobenzene	37
(2) Microagglomeration and removal of ZnS		Sec-butyl xanthate, CuSO ₄ , NaOH	Nitrobenzene	37
(3) Microagglomeration and removal of CaCO ₃		Oleic acid	Nitrobenzene	37
Ilmenite concentrate; removal of complex silicates	Water	Oleic acid, sodium silicate, pH adjuster	Petroleum distillates	38
Iron ores; removal of phosphatic and siliceous matter	Water CaCl ₂ solution	Various fatty acids, bases and acids for pH adjustment	Crude or semirefined viscous petroleum oils	39,40
Marl (CaCO ₃) deposits, removal of siliceous matter	Water	Fatty acid after heat treatment	Kerosene	41
Methyl methacrylate suspension; balling	Light petroleum distillate		Aqueous chloral hydrate solution	9
Shale; removal of CaCO ₃	Water	Sodium oleate	Oxidized crude oil	42
Tar sands, agglomeration of bitumen	Water	Alkali	Hydrocarbons present in sands	43
Tar sands, extraction of bitumen and agglomeration of sands	Petroleum		Water with alkali	44
Tin ore for upgrading	Water	Tall oil, acids and bases for pH adjustment	Viscous petroleum oil	45,46

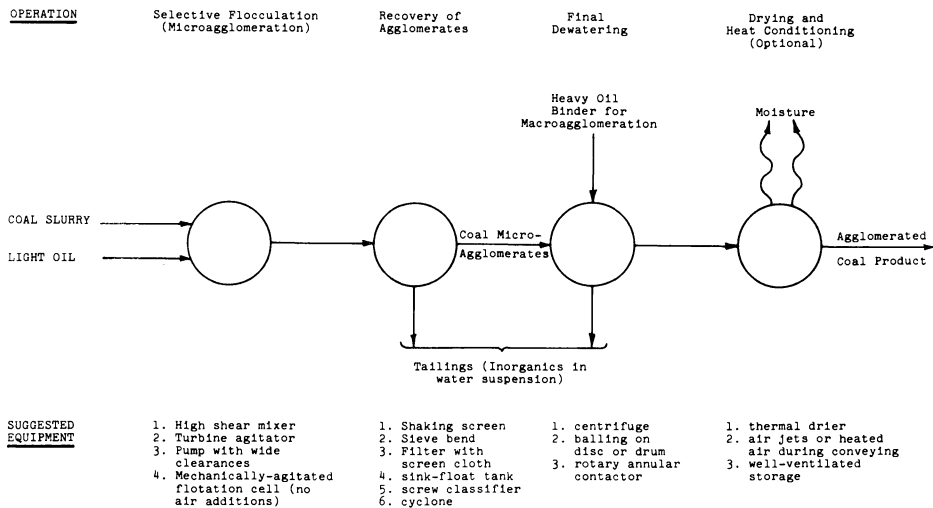


Fig. 8.2. Coal agglomeration flow sheet showing alternative processing steps and types of equipment [6].

beneficiated coal particles with simultaneous further rejection of tailings and moisture and, finally, thermal treatment of the product to remove moisture and/or harden binders. A typical slurry treated by this process would consist of 10 weight % of waste coal particles of minus 200 mesh size consist and 30% of ash-forming impurities. Using perhaps 10 weight % (based on solids) of a light refined oil as agglomerating liquid would result in recovery of more than 90% of the solid carbonaceous matter with a 5 to 10% product ash content.

The most critical step in the coal agglomeration process of Fig. 8.2 is the initial selective agglomeration and the mixing required to accomplish it. Agitation serves initially to disperse the oil phase and secondly to contact the oil droplets and coal particles so that bonds are formed between oil-coated particles. The required intensity and duration of mixing is determined by the oil and coal characteristics and the solids concentration and oil usage. Predispersion of the oil improves this agglomeration step [11]. The range of operating conditions which have been used in this selective agglomeration step includes:

Coal slurry feed (wt. % of solids)	5 to 50
Oil usage (light fuel oil) (% of solids wt.)	2 to 30
Agitator tip speed (m/sec)	~10 to 30
Mixing time	30 sec to several min
Power consumption (kW/m ³)	~10 to 40

A machine developed to remove soot from wash water effluent of oil

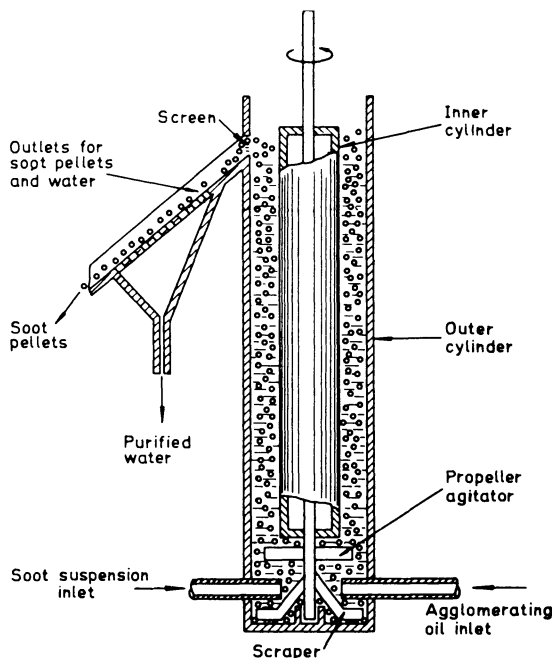


Fig. 8.3. The Shell Penetrating Separator [12] originally developed to remove soot from wash water effluent of oil gasification plants.

TABLE 8.3

Dimensional and operating data for agglomerator shown in Fig. 8.3 [12].

Dimensions:

Outer cylinder	
Length, m	2.550
Diameter, m	0.640
Rotating inner cylinder	
Length, m	2.150
Diameter, m	0.340
Annulus	
Width, m	0.150
Mixing section	
Height, m	0.400
Agitator diameter, m	0.440
Total capacity, l	600

Operating data:

Aqueous soot suspension feed	
Weight % solids	1–3
Average particle diameter, μm	~5
Soot surface area, m^2/g	1000–1500
Agglomerating oil	Bunker C
Operating temperature	70°C
Oil/soot ratio	3.5–4.5
Average residence time, min	2.5–8
Agglomerate hold-up, % vol	7–30
Agglomerate diameter, mm	2–7
Rotor speed, rpm	400–670
Power consumption, HP/m^3	~15

gasification plants is shown in Fig. 8.3. The dimensions of one model of this machine and its operating characteristics are listed in Table 8.3. In this application a completely clear waste water and hard compact oil-bonded soot pellets are obtained [12]. Similar models and later developments of this machine have been used to remove oil from waste water (by adding fine carbonaceous material as a scavenger) and to de-water and de-ash slurries of fine coals [13].

3. Agglomeration by polymeric flocculants

The use of organic polyelectrolytes to bridge between particles in liquid suspension is well known [1,2]. The agglomeration of particles into a floc structure results in faster settling of the suspension and allows the supernatant liquid to be recovered more quickly. The flocculation phenomenon, however, leads to certain problems in the settled material. Flocculated particles tend to stick to each other as they settle and form a loose, bulky layer. The pores in the settled layer are relatively large and its filtration and drainage rate is thus enhanced. On the other hand, the high porosity of the settled layer means that a larger proportion of suspending medium is often retained by the flocculated material than is the case with the unflocculated particles [14].

Techniques have been developed to form more compact sediments (agglomerates) of reduced liquid content in flocculated systems. These techniques combine relatively large amounts (a few pounds per ton of solids) of polymeric flocculants with special mechanical equipment to reduce the moisture content of the separated solids. The agglomerates thus formed contain more interparticle bridges than with lower polymer levels, are able to grow to a larger size permitting easier separation from the liquid phase and are strong and pliable enough to allow entrapped liquid to be squeezed out under mechanical working.

One device, developed in Japan and depicted in Fig. 8.4, makes use of a slowly-revolving (1 m/min peripheral speed) horizontal drum to dewater sludge [15]. The drum interior consists of three sections for pelletizing, decanting and consolidating the solids. Polymeric flocculant is added to the suspension upstream of the drum, together with auxiliary agglomerating agents such as calcium hydroxide or sodium silicate. Voluminous flocs formed ahead of the drum are rolled into denser sediment in the pelletizing section. These are then pushed into the decanting section by a guide baffle where water is removed through intermittent slits in the drum wall. In the final consolidating section, the agglomerates are gently tumbled and rolled into a denser form and water again escapes through wall slits. Product solids then discharge as a low-water-content cake. These cylindrical vessels, known as "Dehydrams", are available in standard sizes up to 3.4 m (11.2 ft) diame-

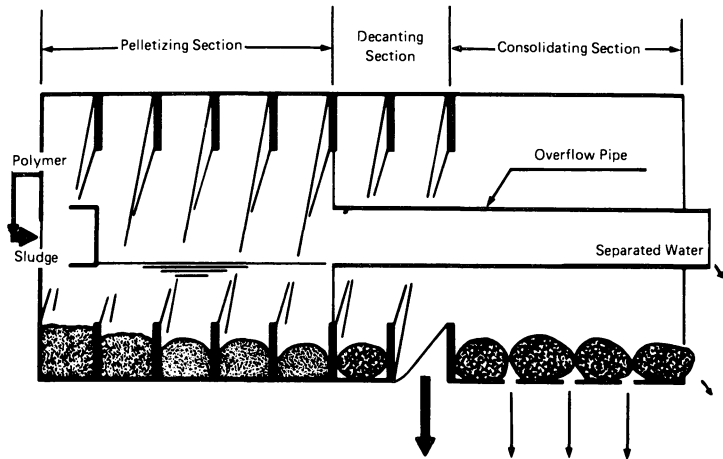


Fig. 8.4. Construction of slowly-revolving horizontal drum for sludge dewatering [15]: the “Dehydram”. (Reprinted with permission of the American Water Works Association from Journal AWWA, Volume 67, Copyrighted 1975.)

ter by 9.2 m (30.2 ft) long with a 5.5 kW (7.5 hp) drive. Typical capacities for a 2.4 m (7.9 ft) diameter by 6.5 m (21.3 ft) long unit are given in Table 8.4 for various sludges, while Table 8.5 provides performance data for the treatment of a number of suspensions.

Another approach to better dewatering of flocculated sludges is through the use of filter belt devices. One type of equipment is shown in Fig. 8.5. A rotary drum flocculator provides a gentle mixing action to ensure optimum floc formation. The flocs then pass to a woven fiber belt for gravity drainage after which the thickened sludge passes under a pressure belt for final dewatering. Performance data with a number of industrial sludges are given in Table 8.6.

TABLE 8.4

Typical capacities for “Dehydram” shown in Fig. 8.4 with various sludges (unit 2.41 m diameter by 6.50 m long, 1.5–3.7 kW driving power).

	t/hr
Gravel waste sludge	7.0–10.0
Shield tunneling bentonite slurry (sludge)	2.0–4.5
Dredged mud slurry (sludge)	1.5–2.4
Water works sludge	0.4–0.6
Automobile factories mixed waste sludge	0.4

Courtesy Ebara — Infilco Co. Ltd.

TABLE 8.5

Performance data for treatment of various sludges using "Dehydram" of Fig. 8.4.

	Type of sludge	Water works sludge	Bentonite slurry sludge in shield tunneling process	Sludge in mixed waste effluents from automobile shops (containing oils and activated sludge)	Sludge in gravel- washing waste water	Sewage sludge (mixed raw sludge)	Sludge from dredging waste water
Raw sludge	Solid concentration, g/l	30—40	150—300	50—70	100—200	40—75	200—250
	Ignition loss of solid matter, wt %, db	15—30	—	40—45	—	40—45	12—21
	Oil content of solid matter, wt %, db	—	—	7—10	—	—	—
Chemicals	Wt % polymeric flocculant, db	0.1—0.2	0.07—0.1	0.25—0.3	0.04—0.05	0.25—0.35	0.05—0.1
	Wt % $\text{Ca}(\text{OH})_2$, db	0	3	0	0	0	2—4
Moisture content in cake, wt %, wb		65—80	46—48	82—86	38—47	79—82	46—64
Turbidity of separated water		50	50	100	100	100	100

Courtesy Ebara — Infilco Co. Ltd.

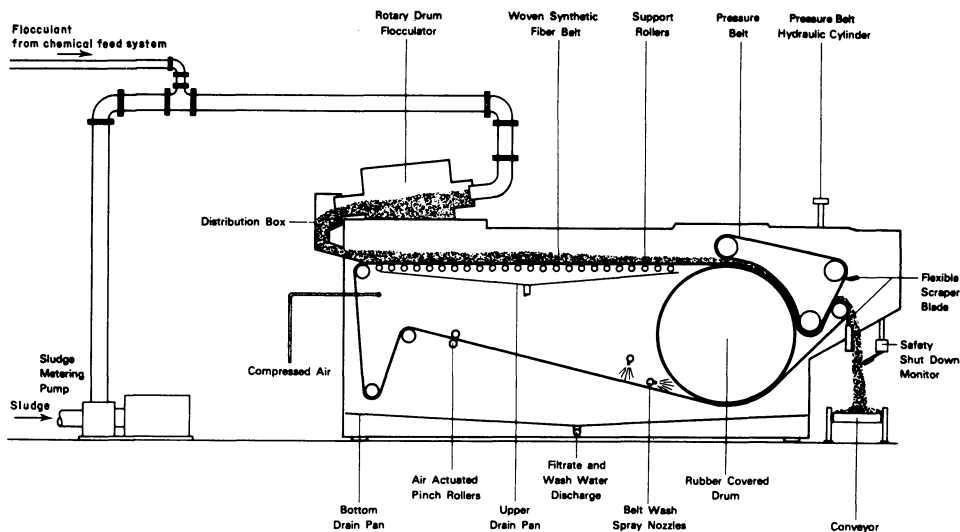


Fig. 8.5. Filter belt device for sludge dewatering: the "Floccpress". (Courtesy Infilco Degremont Inc.)

TABLE 8.6

Performance data for treatment of various sludges using "Floccpress" of Fig. 8.5.

Type of sludge	Feed sludge % dry solids	Output in lbs. per ft. of belt width per hr	Cake % dry solids	Polymer lbs. per dry ton
Municipal sewage				
Raw primary	5—10	161—268	25—35	1.8—4
Anaerobically digested- primary	4—10	168—335	26—36	2.0—6
Mixed primary and waste activated	4—9	87—168	20—28	1.5—6
Anaerobically digested primary and waste activated	3.5—9	134—268	18—28	3.4—8
Aerobically digested	1.5—2.5	54—101	12—18	4.0—8
Raw slaughter house waste	3.5—2.5	134—302	20—30	5.0—8
Lime softening sludge *(Fe/SS-1%)	1.0—15	335—670	55—70	0.4—1
Alum sludge *(Al ₂ SO ₄ + Act. Silica)	3—6	54—85	14—18	1.5—5
Paper mill				
White water sludge *(Al ₂ SO ₄ + Aid)	2.5—4	67—268	20—25	2.0—4
Primary clarifier sludge	4—7	134—335	25—32	0

* Flocculant used to condition sludge.
Courtesy Infilco Degremont Inc.

Choice of flocculant is of prime importance in these processes. Criteria for choosing a flocculant include the degree of floc formation and effect on water clarification, the amount of water contained in the settled and dewatered flocs and the dosage required in terms of cost per unit weight of dry solids. Organic polyelectrolytes provide the best results with many materials and it can be anticipated [16] that a cationic flocculant will be most useful with organic sludges while an anionic or nonionic flocculant will be best for inorganic and mineral sludges.

4. Dispersion in liquid phase

A number of techniques exist whereby solid materials are converted into a liquid form, dispersed as droplets in a second liquid by some suitable means and solidified to a final particulate product. When the starting material is a massive solid, the process is then one of size reduction [17]. When a powder feed is used, size enlargement results.

Variants of this technique differ in the methods of forming the initial liquid phase and dispersing it in a second liquid as well as the resolidification procedure. Prilling into liquid medium (Section 2, Chapter 7) involves formation of melt droplets at elevated temperatures and cooling the droplets to a solid product as they fall through a column of liquid. Similarly powders can be agitated in liquid above their melting temperatures to form droplets and quenched below their melting points to yield solid enlarged particles [18]. Another approach is to dissolve the solid powder in sufficient solvent to render it fluid, disperse by agitation or spray into water and steam-distill the solvent to give solid particles [17]. In the sol-gel process [19,20], small crystallites of various materials are dispersed as an aqueous sol which is then formed into droplets in an organic phase. Gelation into a more solid form occurs in the organic phase, for example by dehydration of the aqueous droplets through the action of higher alcohols in the organic phase.

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Appendix

POTENTIAL SUPPLIERS OF SIZE ENLARGEMENT EQUIPMENT

Following is a list of potential suppliers of size enlargement equipment arranged in the same categories of methods (and chapters) used in this book.

The list is obviously not exhaustive in a world wide sense. Inevitably it reflects the special interests of the author and his North American background.

Unless shown otherwise, the addresses given are in the U.S.A.

Chapter 3. Agitation methods — tumbling agglomeration

Inclined disc and drum agglomerators

- 3.1 Allis-Chalmers, Box 512, Milwaukee, WI 53201
- 3.2 Dravo Corp., Neville Island, Pittsburgh, PA 15225
- 3.3 Eirich Machines Inc., 663 Fifth Avenue, New York, NY 10022
Maschinenfabrik Gustav Eirich, D-6969 Hardheim, Nordbaden, Post-
fach 45, Federal Republic of Germany
- 3.4 Feeco International, Inc., 3913 Algoma Road, Green Bay, WI 54301
- 3.5 Kennedy Van Saun Corporation, Danville, PA 17821
- 3.6 Mars Mineral Corp., P.O. Box 128, Valencia, PA 16059
- 3.7 McDowell Wellman Co., 113 St. Clair Avenue, N.E., Cleveland, OH 44114
- 3.8 The A.J. Sackett & Sons Co., 1701 South Highland Avenue, Baltimore,
MD 21224

Chapter 4. Agitation methods — mixer agglomeration

Pan agglomerators

See entry 3.3

Pugmill agglomerators

- 4.1 International Engineering, Inc., 15 Park Row, New York, NY 10038
 - 4.2 Edw. Renneburg & Sons Co., 2639 Boston Street, Baltimore, MD 21224
- See also entry 3.4

High speed shaft mixer-agglomerators

- 4.3 Bepex Corporation, Rietz Division, P.O. Box 880, Santa Rosa, CA 95402

Equipment for cluster-type powder agglomeration

- 4.4 Blaw-Knox Food & Chemical Equipment Inc., Buflovak Division, P.O. Box 1041, Buffalo, N.Y. 14240
- 4.5 Littleford Bros., Inc., 4143 Airport Road, Cincinnati, OH 45226

Chapter 5. Pressure methods*Piston-type presses*

- 5.1 Pennwalt Corporation, 955 Mearns Road, Warminster, PA 18974
- 5.2 Posey Iron Works, Inc., Mixer and Brick Machinery Div., P.O. Box 239, Lancaster, PA 17604

Roll briquetting and compacting

- 5.3 Bepex Corporation, K-G Division, 10227 Higgins Road, Rosemont, IL 60018
- 5.4 The Fitzpatrick Co., 832 Industrial Drive, Elmhurst, IL 60126
- 5.5 Maschinenfabrik Köppern GmbH & Co KG, Postfach 320, D-4320 Hattingen 1, Federal Republic of Germany
- Vulcan-Kopperrn Inc., United Penn Bank Bldg., Wilkes-Barre, PA 18701

See also entry 3.1

Pellet mills

- 5.6 Process Pelleting Division, California Pellet Mill Co., 1114 E Wabash Avenue, Crawfordsville, IN 47933
 - 5.7 Sprout-Waldron Division, Koppers Company, Inc., Muncy, PA 17756
- See also entry 5.3

Screw-type extruders

- 5.8 The Bonnot Company, 805 Lake Street, Kent, OH 44240
 - 5.9 Fate Root Heath Division, Banner International Inc., P.O. Box 105, Plymouth, OH 44865
- See also entries 4.3 and 5.7

Screen granulators

See entries 5.1 and 5.2

Chapter 6. Thermal methods*Sintering, pelletization and pyroprocessing systems*

- 6.1 Koppers Company, Inc., Pittsburgh, PA 15219
- 6.2 Arthur G. McKee and Company, McKee Building, 2300 Chester Avenue, Cleveland, OH

Drum dryers and flakers

6.3 Ametek, Process Equipment Division, East Moline, IL 61244

6.4 Goslin Division, Envirotech Corporation, P.O. Box 398, Birmingham, AL 35201

See also entry 4.4

Endless belt systems

6.5 Luwa Corporation, P.O. Box 8009, Charlotte, NC 28208

6.6 Sandvik Conveyor Inc., 1650 McBride Avenue, Fair Lawn, NJ 07410

Chapter 7. Spray and dispersion methods*Spray dryers*

7.1 Anhydro, Inc., 165 John L. Dietsch Square, Attleboro Falls, MA 02763

7.2 Komline-Sanderson Ltd., Peapack, NJ 07977

7.3 Niro Atomizer Inc., 9165 Rumsey Road, Columbia, MD 21045

See also entry 6.3

Prilling towers

7.4 HPD Incorporated, 1200 Roosevelt Road, Glen Ellyn, IL 60137

Fluid bed systems

7.5 Aeromatic AG, CH-4132 Muttentz, Farnsburgerstrasse 6, Switzerland

7.6 Fuller Company, P.O. Box 29, Catasauqua, PA 18032

7.7 Glatt Air Techniques Inc., 510 Livingston Street, Norwood, NJ 07648

See also entries 6.3 and 7.3

Spouted bed systems

7.8 Coating Place, Inc., Box 248, Venona, WI 53593

7.9 Lakso Company, Inc., 44 Mead Street, P.O. Box 929, Leominster, MA 01453

Pneumatic flash dryers

7.10 Combustion Engineering Inc., 200 West Monroe Street, Chicago, IL 60606

See also entries 6.3, 7.1 and 7.2

Chapter 8. Agglomeration in liquid media*Sludge dewatering devices*

8.1 Ebara-Infilco Co., Ltd., The Palaceside Bldg., 1-1, 1-Chome, Hitotsubashi Chiyoda-Ku, Tokyo, Japan

- 8.2 Infilco Degremont Inc., Koger Executive Center, Box K-7, Richmond, VA 23288
- 8.3 The Permutit Company, Inc., E. 49 Midland Avenue, Paramus, NJ 07652

Immiscible liquid technology

- 8.4 American Minechem Corporation, Coraopolis, PA 15108
- 8.5 Arcanum Corporation, P.O. Box 1482, Ann Arbor, MI

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